THE EFFECT OF LOCAL RECYCLED WASTE MATERIALS ON THE PHYSICAL BEHAVIOR AND PERFORMANCE OF JOINT FILLING AND WATERPROOFING ASPHALT

BY

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LIST OF ABBREVIATION

ANOVA	:	Analysis of Variance
SBS	:	Styrene Butadiene Styrene polymer
CKD	:	Cement Kiln Dust
EVA	:	Ethylene Vinyl Acetate Polymer
HOFA	:	Heavy Oil Fly ash
LMD	:	Limestone Dust
OS	:	Oil Sludge
Mod-EVA	:	Sulfur modified Ethylene Vinyl Acetate
SP	:	Softening Point
FP	:	Flash Point
BS	:	Bond Strength
BST	:	Bond Strength Test
F_{value}	:	Statistic Fisher Value
B	:	Percent Composition of SBS by weight asphalt
C	:	Percent Composition of CKD by weight of Sulfur/sludge/polymer modified asphalt
H	:	Percent Composition of HOFA by weight of Sulfur/sludge/polymer modified asphalt
L	:	Percent Composition of LMD by weight of Sulfur/sludge/polymer modified asphalt
G	:	Percent Composition of OS by weight of asphalt
S	:	Percent Composition of Sulfur by weight of asphalt
V	:	Percent Composition of Mod-EVA by weight of asphalt

THESIS ABSTRACT

Name: MUHAMMAD ABUBAKAR DALHAT

Title: THE EFFECT OF LOCAL RECYCLED WASTE MATERIALS ON THE PHYSICAL BEHAVIOR AND PERFORMANCE OF JOINT FILLING AND WATERPROOFING ASPHALT

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Waste materials recycling has been the logical and widely accepted means of conserving the diminishing global natural resources, this is as a result of increased scarcity of raw industrial materials, coupled with environmental hazard of most of the waste. For the same reason there is an increasing interest in studies focusing on finding alternative material and economical material sources. In this work, the effect of different waste material fillers (namely: Lime dust, Heavy oil fly ash and cement kiln dust), sulfur, oil sludge on the physical properties and performance of roofing and waterproofing asphalt has been examined. Physical and performance testing (Bond strength test, Softening point test, Ductility test, Penetration test, Viscosity and Flash point test) were conducted on the modified asphalt mastic, and finally the results were analyzed statistically and properties modeled in terms of additives composition. Some selected blends were compared to conventional roofing asphalt mastic blend. Results show promising potential alternative and cost effective material composite having least amount of asphalt component.

ملخص الأطروحة

ألاسم: محمد أبو بكر طلحت

العنوان : أثر مواد النفايات المعاد تدويرها على سلوك وأداء أسفلت تعبئة الشقوق و اغشية عزل الماء

فسم : الهندسة المدنية

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تدوير نفايات المواد كانت ولا تزل وسائل منطقية ومقبولة على نطاق واسع للمحافظة على الموارد الطبيعية ألمتناقصة وذلك نتيجة لزيادة ندرة المواد الصناعية ألخام إلى جانب المخاطر البيئية لمعظم النفايات. هناك اهتمام متزايد بالدراسات التي تركز على إيجاد مواد بديلة ومصادر لمواد اقتصادية. هذا البحث يدرس تأثير مختلف مواد حشوه الأسفلت من النفايات والتي تشمل غبار الجير ورماد الزيت الثقيل المتطاير وغبار الأسمنت والكبريت وحمأة خزانات النفط على الخواص الفيزيائية وأداء اغشية الاسفلت وموانع تسرب المياه. أجريت الاختبارات الفيزيائيه والأداء (والتي تشمل اختبار الإختراق واللزوجة والإستطالة ودرجة التميع والوميض وقوة الالتصاق) على معجون الأسفلت ألمنتج وأخيرا تم تحليل النتائج إحصائيا وتم نمذجة الخصائص باستخدام نسب ومكونات مواد الحشوه وتمت مقارنة خصائص الخلطات مع تلك المستخدمة تجاريا ولقد أظهرت النتائج مركبات واعدة وبديلا محتملا وفعال التكلفة وتحوي أقل قدر من عنصر الأسفلت.

CHAPTER 1

INTRODUCTION

Traditionally, asphalt-based roofing and waterproofing products were made from air blown asphalt, but as roofing chemistry became more sophisticated, asphalt mastic[1] of various formulations with different viscosity ranges, physical and mechanical properties (for horizontal and vertical applications) were developed for specific uses from a regular asphalt/bitumen by chemical and mineralogical contents modification, such as pourable sealers (pitch pocket mastics), elastomeric sealants (mastics for high movement joints and terminations) etc. This is due to the fact that the improvements achieved on the bituminous material's durability and extensibility (especially at lower temperatures) by adding polymers, results in modified material with superior physical property that surpass any other alternative material for the same cost.

Sufficient attention has not specifically been given to asphalt constituent of roofing materials at the earlier stage, component such as granular coating and reinforcement fibers were the main focus for improving serviceability performance [2, 3, 4, 5]. Now the asphalt polymer modification is well explored, understood and lots of works were published on the subject [10, 11, 12, 13, 14, 15]. Apart from polymer, another major additive which also dictates the final performance of this waterproofing mastic asphalt, is the mineral filler. Studies have been conducted to investigate the effect of different mineral fillers on certain properties of asphalt binder [17, 18, 19], but nearly all of these works are directed towards behavior and durability of asphalt concrete pavement ‘AC’, not roofing or water proofing application. And also there is a monopoly in the selection of mineral filler type for roofing purpose [1], which is limestone dust. The possibility of an alternative filler additive better or equivalent to limestone in terms of serviceability performance of waterproofing asphalt has not been explored so far.

Sulfur extended asphalt has been used in ‘AC’ to its improved resistance to rutting and storage stability [21, 22, 23], but its roofing/waterproofing performance implications was not addressed. It was also used for roofing applications [20], but with as just a mere extender without fire safety hazard assessment such as the ASTM specification test which is D0092 (flash and fire point test) or D2822 (standard specification for asphalt roof cement) which has not been adopted at the time.

There is also an increased need and pressure globally for a shift towards green construction approach. By green it means the consumption of non-renewable virgin natural resources should be minimized through the recycling of waste materials, this will in turn ease and facilitates the waste disposal process. Also the amount of energy require for processing virgin raw material is almost always less than that required for recycled materials, as a result, the amount of environmental carbon foot print will be less. So in this research five different waste materials namely; Heavy oil fly ash ‘HOFA’, Cement kiln dust ‘CKD’, Lime dust ‘LD’, Tank bottom oil sludge ‘OS’ and sulfur were employed.

The influence of sulfur, Heavy oil fly ash 'HOFA', Cement kiln dust 'CKD', oil sludge and Limestone dust on the physical properties of asphalt namely softening point, ductility, penetration, flash point, viscosity and bond strength were studied.

1.1 PROBLEM STATEMENT

The global asphalt demand which has been estimated to be over 100 million metric ton in 2007, results from its various uses and application growth that mainly include road pavement construction and roofing/waterproofing appliances [36]. More than 85% of the asphalt produce goes in to road construction, this, in addition to feature prediction of further rise in demand from this area of application present a fierce competition and treat to the stability and sustainability of the asphalt supply and finished product prices for waterproofing and roofing application, which is now believed to be among the recent fastest growing market with an approximate demand of 11% of the total global asphalt demand.

Another global concern that has to do with our environment is natural raw material preservation, which is completely a function of how we manage these resources, be it renewable or non-renewable, but especially the non-renewable ones that can only be preserved in accordance to how fast we decide to exploit them. This brings us to the unavoidable issue of waste disposal and recycling. All waste material must be disposed of through means and manner that pose less or zero treat to the environment, and will be an added advantage if alternative disposal process by using the waste for other useful application can be found, a process known as 'recycling'.

The evolution of asphalt role and end product type for roofing and waterproofing uses is resulting to various challenges that require new investigative approach in order to improve product durability and performance and to also optimize material usage for cost effectiveness. For instance, at the early stage in the utilization of asphalt for roofing purposes, it was widely resolved and accepted that air-blown asphalt (due to its extra hardness) is the perfect choice for the job. But it was later realized and understood with time that air-blown asphalt is short in flexibility and durability when compared to polymer-modified asphalt. Thus given way to polymer modified asphalt's dominance in the field. But before this science was actualized and perfected, lots of changes in research approach were have to be made. And now, another step forward is needed, which is a more close investigation of other possible additives that has the potential of improving the durability and performance of roofing and waterproofing asphaltic component apart from the polymer.

1.2 OBJECTIVES

The main objectives of this work are: to conduct a study on the influence of these additives (HOFA, CKD, Limestone dust, oil sludge, modified-EVA and sulfur) on the physical behavior of roofing asphalt, based on laboratory experimental result of samples prepared from waste modified-blends and previous research works; to establish general trend, report observations based on analogies and previous literature; to explore the possibility or suitability of using this waste (HOFA, CKD, Limestone dust, Oil sludge and sulfur) as material substitute for superior quality and cheap product; And to investigate a new means of assessing the bond strength of the mastic asphalt at semi-finished product level.

1.3 SIGNIFICANCE OF STUDY

Cement companies are among the top largest expanding enterprises in Saudi Arabia and Arab counties, their annual production are in order of millions of tones, for each 1-tone of cement produced, approximately 1.5-1.7 tons of raw material has been processed. The rest has to go as waste (cement kiln dust '**CKD**'). This waste is costing the companies millions of Saudi Riyals annually for proper disposal. Even though in the united state the cement dust has found many applications such as agricultural soil enhancement, pavement base soil stabilization, waste water treatment etc such practices are not common in the kingdom.

The **sulfur** is from a processed crude oil refining industrial waste of which commercial value will only increase if its potential benefit is explored and brought to light. This will help reduced the cost accrue from waste disposal in the oil industry within the kingdom. Also the **bottom oil sludge** is another typical waste material having health and economic implication with regard to disposal issue, if positive results should be obtained as regard to its asphalt modifying property, then two problems will be simultaneously addressed; the quantity of asphalt could extended and material properties could be improved.

Additionally, heavy oil fly ash '**HOFA'** is a waste by-product of heavy oil combustion from electric power thermal stations. It contains some heavy metal oxides like vanadium and nickel [9], which are carcinogenic in nature (when inhale or consume by any means to a certain level will cause cancer and many health ailments). So proper disposal is not an option but a necessity, and the suitable economical means of disposing it will be to recycle it.

It is obvious that limestone (lime dust) is not a concern in the kingdom **(Saudi Arabia)** when it comes to availability and cost issue, especially in the eastern province where the largest waterproofing and cement industries are located. But both lime-dust and CKD are waste materials which has as much environmental impact and economic abundance as the limestone its self, specifically the CKD. The same reason that attract asphalt product waterproofing industries to locate to the province also lead cement companies to establish here (availability and proximity of raw material). The quantity and rate at which the limestone is engulf (in the production of cement alone) is two-fold as long as the cement waste (Lime-dust & CKD) produced, which is more than 25% of the initial raw material cannot be recycled to relieve the same natural material source.

CHAPTER 2

LITERATURE REVIEW

Most of the previous work in this area focuses on the polymer effect on the asphalt component [12, 13, 14, 15]. Performance test and specification for asphalt in roofing applications are only restricted to final product, these test include tensile strength test, flexibility test, impact resistance test, adhesion to substrate and protective coatings test etc [2, 3, 4]. Some other tests are employed by individual factories, but are not standardized [37]. Possible means of evaluating the service performance of unprocessed modified asphalt for roofing and waterproofing functions are being explored [7].

2.1 Asphalt Shingles

Asphalt singles: roofing materials which either utilizes organic felts (wood and paper pulp products) or glass fiber mat reinforcement (also called substrates) that are saturated with oil-rich asphalt in order to be rendered water-tight, and which is further embedded with protective granules (either ceramic or mineral aggregates) by means of more viscous asphalt top coating, are termed ***asphalt shingles***.

The oil-rich saturant asphalt serves as a preservative, water proofing, as well as adhesive agent. The granular materials which are bonded to the surface of the more viscous asphalt coating provides various aesthetic colors, shield to the asphalt component from direct sunlight exposure which helps in minimizing asphalt deterioration by photo-degradation (ultra-violet light), in addition to increase fire resistance. The coating asphalt basically contains finely divided minerals such as silica, slate dust, talc, micaceous materials etc as stabilizers or fillers.

Different types of asphalt shingles were patented in the last few decades, each having a unique improved property, it will be seen that the earlier works focus more on **Substrate** and **granular protection** improvement rather than the coating **Asphalt**:

A process of improving the bond between the roofing granules and the coating asphalt is among the popular patented work in this field [2, 3]. This method suggest spraying a controlled amount (25%-75% surface coverage) of a thermoplastic non asphalt adhesive having a low viscosity at temperature between 150^{o}C and 260^{o}C, which is capable of forming a moisture resistance bond coupled with a bond strength greater than the coating asphalt (typically ranging from 300psi to 2100psi) to the surface of the hot asphalt prior to the application of the roofing granules. It was established through the means of a dry rub test (ASTM 4977-89) that the asphalt roofing granule adhesion is improved by more than 2 fold, also the wet rub-loss of roofing granules is reduced by a factor greater than 300times in a 1-day wet rub-test, and by 6times in seven days wet rub test.

The incorporation of a thermoplastic polymer fiber web having a higher tensile strength than the organic felt or glass fiber reinforcement at the lower part of the shingle in order to improve its impact resistance to punching and wear tear (for example due to hailstorm etc) has been proposed [4]. The web have two components with difference in melting point of atleast 28^{o}C, this allows the web to be thermally fused within the hot lower coating asphalt of the shingle while developing a strong bond without shrinking, which is capable of dissipating impact energy. This eliminates the necessity for using modified asphalt with a better impact forbearance instead of conventional asphalt. A lab test ‘standards for impact resistance of prepared roof covering materials’ (UL 2218) is carried out to ensure that roofing material having a web exhibits a 2times impact resistance relative to the usual roofing shingle as a control.

A new layer ‘protective coating’ was later introduced to the previous invention [5]: a thin film adhesive (typically flexible ethylene-vinyl acetate copolymers, ethylene-vinyl acetate copolymers modified with styrene-butadiene-styrene (SBS) or tackified polyethylene) is applied to the hot coating asphalt (100% coverage) with the view to minimizing the loss of granule due to hail impact and effect of weathering, it also provides additional moisture barrier. A granular adhesion testing which simulates effect of weathering and hail show 44% loss of granules for a normal shingle while those having a protective coating of ethylene-vinyl acetate copolymers, SBS modified ethylene-vinyl acetate copolymers and polyethylene shows a loss of 3%, 5% and 2% respectively.

2.2 Polymer in Roofing Asphalt

A large number of polymer modified asphalt/bitumen waterproofing product are commercially available, but because of market strategy and necessity, comparatively little work is published on the subject. Some of the work reported so far, are highlighted below.

A means of characterizing a polymer-modified bitumen roofing membrane using rheometer oscillatory shear test ‘ROST’ has been proposed [7]. Three polymers (namely:

high density polyethylene ‘HDPE’, a metallocene catalyzed linear low density polyethylene (m-LLDPE), and a recycled EVA copolymer) were employed for this study. It is demonstrated that a relaxation spectrum index ‘RSI’ which could be obtained from the G’ and G’’ ‘ROST’ plot, can be used as standard parameter for designating the minimum amount of polymer required to ensure a continuous polymer-rich phase (phase inversion), and also as a measure of the blend elasticity. The tacking/adhesive property of modified-bitumen as a function of G’ (which should not be less than 3.3x105) could also be measured by ‘upper plate rheometer separation’ test, under a controlled condition.

A poly-urethane-modified bitumen (or asphalt) having superior property than a conventional one has been determine [10], which could be use as coating compound, adhesives and joint sealants in roofs. The modification is achieved through combining a primary and secondary plasticizers (namely: butyl-urethane-formaldehyde-carbamic acid ester resin and 2,3-dibenzyltoluene) with bitumen in equal proportion, due to incompatibility of bitumen with polyurethane.

Another work on asphalt impermeable moisture membranes is a patented means of producing a poly-butadiene urethane-polymer-modified bitumen roll sheet moisture barrier material for concrete structures [11]. The sheet composed of a non-heat shrinkable thin surface layer (e.g olefinic resin, polyethylene or polyester), A layer of an asphalt-containing mineral filler (typically a calcium carbonate ‘$CaCO_3$’ particulate ranging from 5%-25% by weight composition of asphalt) along with a urethane polymer (reaction product of poly Isocyanate with a long chain poly butadiene hydrocarbon). It was observed that 3% to 23% of hydroxyl-terminated poly butadiene together with 0.4% to 2.8% Isocyanate yields an asphalt material with an adequate structural stiffness and flexibility (within -12^{o}C to 53^{o}C), which has sufficient tackiness at lower temperature as well as non-fluidity at upper temperatures.

The roofing properties of an MDI-PEG (methylene-diphenyl diisocynate-polyethene glycol) modified bitumen has been studied and compared to a conventional (12wt.%) SBS (styrene-butadiene-styrene) altered bitumen roofing material [12]. Viscous flow readings at 60^{o}C for a 8wt.% MDI-PEG added sample obtained from an RS-150 rheometer show a slightly greater value after 15days curing. But Ring and ball softening test (ASTM D-36) and Oscillatory shear tests (from 0.01 to 100 rad/s at -20^{o}C) indicate a relatively inferior thermal-flow and thermal-viscoelastic behavior at high and low service temperature respectively. Constituent composition and micro-structural findings from MDSC (modified differential scanning calorimeter) and AFM (Atomic force microscope) respectively reveals new microstructure coupled with a more compact asphaltic regions, implying a bitumen-base composite with a low shear rate viscosity and less temperature susceptibility (with increasing MDI-PEG content) compared to a neat bitumen. It was later established through FT-IR analysis that the viscosity time-improvement is due to formation of higher molecular weight compound from chemical reaction of polymer isocyanate group (NCO-) with hydroxyl (-OH) and amine (-NH) groups in the resin fraction of the bitumen to form urea bond crosslink [13]. Furthermore, the curing rate could be speed-up by adding some amount of water molecule during the mix blending, but this will be at the expense of long term increase in viscosity, due to prevention of further inter polymer-bitumen urea bond by water-exhaustion of the unreacted NCO-group, forming intra-polymer molecular urea-bond instead.

The impact of a hydrogen-peroxide-treated (ozonized) PVC pipe waste on bituminous mastic has been reported [14]. Mastic samples were prepared from an SBS (20%wt. to 30%wt.) modified bitumen, with varying contents of coarse and micronized H_2O_2-treated PVC particulates (60%-70%wt.), along with limestone dust (7%-15%wt.). The ozonized PVC waste demonstrates a better performance in terms of improved viscoelastic properties (as indicated by DMA-Dynamic Mechanical Analysis, rheometer test result etc.). This is attributed to a lower molecular mass, a much rougher and porous surface nature of the treated particles as shown by evidence from a UV-visible spectrometer and SEM (Scanning Electron Microscope), which leads to a consistent and better particle-bitumen anchorage. A roof mastic composition of treated coarse and micronized PVC waste, Isocyanate waste, limestone dust, anti-oxidant, rosin and SBS modified bitumen that satisfied the Indian specification (IS 1195-90 Bitumen Mastic for Flooring) was finalized.

Furthermore, the modification of waterproofing asphalt by PVC packaging waste has been conducted [15]. Samples from asphalt containing (0-10%wt. of asphalt) PVC waste were subjected to low temperature flexibility test, elongation and tension test, alkali and acid resistance test, softening point, ductility and penetration test during a 12month aging cycle period. The results reveal positive performance improvements. This is connected to the FT-IR (Fourier-Transform Infra-Red) findings that shows negligible difference in the locations and magnitude of peaks in absorption band between the modified and the neat asphalt, which insinuates a compatible physical interaction among the PVC waste and the light oil asphalt constituents. Additional microscopic image shows the emergence of disperse and continuous polymer-rich microstructure with increasing polymer content.

2.3 Mineral Filler Stabilizers

The influence of granite dust on the composition and properties of asphalt upon their contact at 160^oC has shown considerable decrease, as well as an increase in penetration and softening point of the asphalt respectively [16]. Further investigation reveals at high concentration (95% content) of large surfaced area mineral filler, all asphalt components are absorbed, resulting to a non-monolith (unstable) black colored powdered mixture. This is contrary to the behavior of a relatively small surfaced area asphalt concrete aggregate, which usually exhibit a maximum stability within 94% to 96% aggregate content.

Mineral filler rheological property effect on different types of bitumen was examined [17], two kinds of minerals namely; calcite and quartz were used for this study. Dynamic shear (DSR) and bending beam (BBR) rheometry tests were carried out on sample bitumen mastic containing filler ranging from (0% to 28%) by volume of bitumen. The stiffening effect is discovered to be filler dependent as well as bitumen dependent. Another bending beam rheometric (BBR) analysis of low temperature physical hardening of hydrated lime and calcium carbonate particulates was made [18], which reveals an insignificant effect of the fillers on the low temperature hardening.

Influence of filler minerals on the aging resistance of bitumen was reported [19], two minerals, calcium carbonate and hydrated lime were compared. Test specimens were made from a single asphalt concrete mix using four (4) filler volumetric composition (0%, 0.5%, 1%, 1.5%). A new tensile test was conducted on samples aged for 0, 2 & 7 days. Softening point test, viscosity and penetration test were carried out on asphalt recovered from hydrated lime mixed sample. It was established that aging resistance increase with filler content, and that the hydrated lime has a much lesser brittle stiffening effect than the calcium carbonate.

2.4 Sulfur Modified Asphalt

One of the major applications of sulfur is in rubber industry, where rubber or related polymers are converted in to more durable materials through a chemical process known as *vulcanization*, a process of heating polymer with a controlled amount of sulfur to form crosslink (bridges) between individual polymer chains. Depending on the nature and characteristics of the polymer, this reaction occurs at a certain temperature known as the vulcanization temperature, which lies above 130^{o}C for a neat asphalt/sulfur blend. It will be important to note that the amount of sulfur required depends on the intended role which the sulfur elemental molecules are expected to play, Sulfur in sulfur-asphalt blends has been found to occur in three different forms: (i) chemically bonded, (ii) 'dissolved' in asphalt, and (iii) crystalline sulfur which generally exists in the form of discrete tiny particles dispersed in asphalt. So the role played by the sulfur constituent is a function of the mixing temperature, the period of mixing, the nature of the asphalt itself, and the quantity.

A built-up roofing composite consisting of two or more felt layers saturated with asphalt containing 10% to 55% sulfur was patented [20]. Even though the product was design with fire resistance in mind, the flash and fire point of the sulfur base asphalt constituent has not been reported.

The impact of sulfur on the rheological and morphological properties of asphalt modified with SB (styrene-butadiene) copolymer has been examined [21]. Two polymers SB and SBS were used. Fluorescence microscopic evidence shows that 5% and 12% SB-modified asphalt blends containing 3phr (part in hundred) sulfur exhibits a relatively more homogeneous polymer-asphalt morphology, with the larger component in continuous phase. This is coupled with mechanical and heat storage stability. Even though the SB-asphalt blend has inferior rheological property compared to the SBS-blend, an SB-blend with better quality matching the SBS's is obtained by adding some amount of sulfur.

Aging effect on storage stable SBS/Sulfur-modified asphalt was investigated [22]. Bending beam rheometer (BBR) and morphological studies of aged and un-aged SBS & SBS/Sulfur-modified asphalt shows that, after aging the SBS/Sulfur mix exhibits an improved low-thermal creep property along with increasing compatibility respectively. The storage stable SBS/Sulfur mix is discovered to be more susceptible to oxidative aging and dynamic shear, which are dependent on the structural characteristics of the SBS.

Similar studies of the effect of sulfur on storage stability and compatibility of tire rubber (TR) modified asphalt has been reported [23]. The vulcanized blends show low temperature penetration susceptibility (TPS) along with high penetration index (PI) values which implies an enhanced thermal susceptibility. They also seem to display much higher low temperature ductility (1% sulfur). Marshall stability and rutting test shows that the 1% sulfur containing mix to possess higher stability as well as rutting resistance respectively. Moreover 160^oC was observed to be the probable vulcanizing temperature of the composite.

Other studies alike, [24, 25] have reported the same use of sulfur for improvement in temperature storage stability, compatibility etc

2.5 Cement Kiln Dust (CKD)

Cement kiln dust is created in the kiln during the production of cement clinker. The dust is a particulate mixture of partially calcined and un-reacted raw feed, clinker dust and ash, enriched with alkali sulfates, halides and other volatiles. These particulates are captured by the exhaust gases and collected in particulate matter control devices such as cyclones, bag-houses and electrostatic precipitators. Several factors influence the chemical and physical properties of CKD. Because plant operations differ considerably with respect to raw feed, type of operation, dust collection facility, and type of fuel, the use of the terms typical or average CKD when comparing different plants can be misleading. The dust from each plant can vary remarkably in chemical, mineralogical and physical composition [26].

However, to provide a general reference point, a typical dust composition as reported by the US Bureau of Mines is given in *Table 1* below.

Table 2.1: Typical chemical composition of CKD

Constituent	% by weight	Constituent	% by weight
$CaCO_3$	55.5	Fe_2O_3	2.1
SiO_2	13.6	KCl	1.4
CaO	8.1	MgO	1.3
K_2SO_4	5.9	Na_2SO_4	1.3
$CaSO_4$	5.2	KF	0.4
Al_2O_3	4.5	Others	0.7

At many facilities all or a major portion of the dust is recycled back into the kiln to supplement the raw feed. Other facilities market their CKD for beneficial commercial uses. For CKD not returned to the kiln system, the most common reasons are equipment limitations for handling the dust and chemical constituents in the dust that would be detrimental to the final cement product or would make the product non-compliant with applicable consensus quality standards. The fraction of the CKD that is not returned to the kiln or otherwise beneficially used is placed in landfills or wasted.

Cement kiln dust produced in a local cement production plant in Saudi Arabia, along with fly ash obtained from combustion of heavy fuel oil in a local power generation plant were utilized as waste materials blended with ordinary Portland cement at various ratios [27]. These blends were tested for their water requirements for normal consistency, initial setting times, and compression and tensile strengths, and were compared to those of Portland cement. Test results show that satisfactory mechanical strength (a minimum of 94% of compression strength of ordinary Portland cement) can still be achieved in blends utilizing 90% cement and not more than 4% fly ash. Adequate mechanical strengths (a minimum of 80% of compression strength of Portland cement) were achieved in blends utilizing as little as 70% of cement when only kiln dust was blended. CKD has been used in many other applications ranging from sub-grade soil stabilization for highway construction [28], to waste treatment and cement replacement [29].

2.6 Limestone Dust (LMD)

The limestone dust (LMD) is also another waste material that mainly comes from cement manufacturing. The quarrying of the raw material (limestone) and its initial processing such as milling, grinding and feeding generate large amount of the limestone dust, these waste are usually capture using the centrifugal collection system. The main constituent of LMD are not different from that of the original material source (limestone), mainly, Calcium ‘Ca’, Carbon ‘C’ and oxygen ‘O’ that made up the rock complex; calcium trioxocarbonate ($CaCO_3$).

Limestone powder waste ‘LPW’ was used along with wood saw waste ‘WSW’ to produce a low-cost light weight composite building material [30]. The results show that the effect of high-level replacement of WSW with LPW does not exhibit a sudden brittle fracture even beyond the failure loads, indicates high energy absorption capacity, reduces the unit weight dramatically and introduces smother surface compared to the current concrete bricks in the market. The potential environmental and performance benefit of using Limestone dust in unbound and hydraulically bound road base materials has also been reported [31]. Samples containing 5, 10, 15 and 20% limestone dust were subjected to tri-axial testing. The results show that control samples, which contain no limestone dust, were the strongest and samples containing 20% dust were the weakest, both presented an unbound material behavior. However, it was decided to reclaim the reduction in the strength of the samples containing 20% limestone dust by adding 4 and 9% pulverized fuel ash (PFA) activated with 1 and 2% hydrated lime respectively as the aim of the project is to use high amount of limestone dust. Interestingly, the results show an increase in new materials resilient moduli of approximately 420%.

2.7 Bottom Oil Sludge (OS)

The bottom oil sludge (OS) is a waste material which accumulates on the bottom of crude oil separation vessel or storage tanks with time. Its constituent will depend on the inherent impurities from the original crude, but mostly it consist of rust debris coming from the inner surface of the transport medium pipe lines, the impurities from oil source which might include some soil etc

An environmental friendly disposal techniques of tank bottom oil sludge has been investigated [32]. Environmental testing includes determination of heavy metals concentration; toxic organics concentration and radiological properties. Solidified sludge mixtures and road application sludge mixtures were subjected to leaching using the toxicity characteristic leaching procedure (TCLP). Tank bottom sludge was characterized as having higher concentrations of lead, zinc, and mercury, but lower concentrations of nickel, copper and chromium in comparison with values reported in the literature. Natural occurring radioactive minerals (NORM) activity values obtained on different sludge samples were very low or negligible compared to a NORM standard value of 100 Bq/g. The apparent lack of leachability of metals from solidification and road material sludge applications suggests that toxic metals and organics introduced to these applications are not readily attacked by weak acid solutions and would not be expected to migrate or dissolved into the water. Thus, in-terms of trace metals and organics, the suggested sludge applications would not be considered hazardous as defined by the TCLP leaching procedure. In another previous similar research [33], the findings are not different: None of the road base samples was hazardous according to the TCLP analyses; But leachates from all the samples were toxic according to the 48-hour acute toxicity test; none of the samples had unacceptable levels of NORM. There is no disagreement whatsoever between the two researches, but just an additional 48-hour toxicity test in the later. And the characteristic of sludge from different oil source is expected to differ.

2.8 Heavy Oil Fly Ash

Heavy oil fly ash (HOFA) used in this study is by-product of fuel combustion process, which includes diesel and cracked fuel. Large quantity of HOFA is produce through the burning of these fuels at thermal power generation plants. HOFA is typically a black powder type of waste material containing mainly carbon. Typical chemical constituent analysis of HOFA is shown on the Table 2. The amount of each element can vary depending upon the source of the heavy oil fly ash.

Table 2.2: Main Chemical Constituent of HOFA

Element composition of HOFA [35]	
Element	**Weight %**
Carbon	92.5
Magnesium	0.79
Silicon	0.09
Sulfur	5.80
Vanadium	0.61

The re-use of oil fly ash (HOFA) for wide variety of purposes can be seen from recent and old literature, it has been studied as an adsorption medium for CO_2, the so called carbon capture. Its potential as a filler reinforcement in light density poly-ethylene (LDPE) polymer composite has been reported [34], results show an improvement in rheological properties of the modified LDPE. Also, the patented means of improving the performance of an asphalt binder and concrete with use of OFA has been disclosed [35]. The OFA- asphalt mixes prove to be within the asphalt performance grade limits with potential improvement in durability and strength.

CHAPTER 3

METHODOLOGY

The mineral filler and extender materials are the main focus of this work, different test samples were prepared from mineral filled sulfur/sludge modified-asphalt, the fillers are heavy oil fly ash ‘HOFA’, cement kiln dust ‘CKD’ and limestone dust ‘LMD’. These blends are tested in accordance with ASTM D113, ASTM D5, ASTM D36, ASTM D92 and ASTM D4402. The properties variation with change in the composition of these waste are evaluated using appropriate charts. Statistical analysis (analysis of variance) has been conducted to ascertain the effectiveness or otherwise of the various additives in combination, with each playing a different role of either extender (sulfur and sludge) or filler (CKD, LMD, HOFA). Regression models of the various properties as the function of the additives were generated. All the statistical evaluations including the regression analysis were conducted using mini-Tab statistical application software.

The scheme of the research work is demonstrated by the flow chart below in *Figure 3.1*. Starting from literature review, followed by materials (polymers, asphalt, waste materials) collection and characterization, then mastic blending or mixing in controlled environmental condition, physical and performance testing, finally result analysis and modeling. Some selected blends were tested for bonding tensile strength performance.

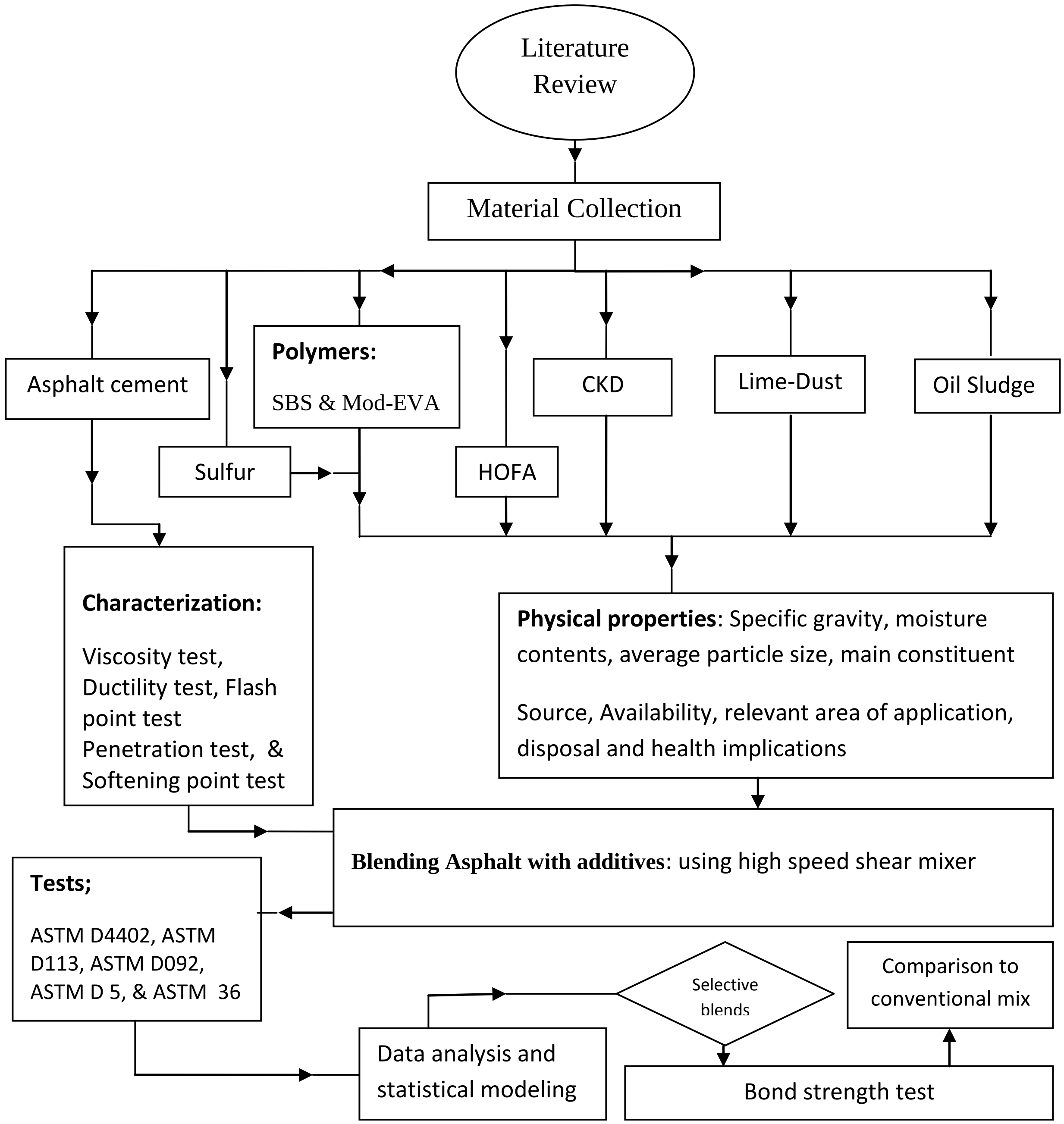

Figure 3.1: work flowchart

3.1 Materials

3.1.1 Asphalt

The asphalt used in this study is the only local available grade, and it is obtained from AL-YAMAMA road construction company. It has the following physical properties (see table 3.1).

Table 3.1: Asphalt Physical Properties

Property	**Magnitude**
Ductility (cm)	150^{+}
Penetration (dmm)	67.2
Softening point (^{o}C)	52
Flash point (^{o}C)	342
Viscosity (cP)	575

3.1.2 Waste and Additive Materials

The Oil sludge is obtained from Rastarona storage tanks, Saudi Aramco oil industry. While the cement kiln dust (CKD) and limestone dust (LMD) byproducts of limestone quarrying and cement manufacturing are obtain from the same source as the asphalt. The sulfur is obtained locally from Saudi Aramco oil company. The heavy oil fly ash (HOFA) is collected from Rabiq thermal power station located within the western province of the kingdom (Saudi Arabia).

3.1.3 Polymers

SBS: Poly(styrene-butadiene-styrene), or SBS, is a hard rubber that's used for things like the soles of shoes, tire treads, and other places where durability is important. It's a type of copolymer called a block copolymer. Its backbone chain is made up of three segments. The first is a long chain of polystyrene, the middle is a long chain of poly-butadiene, and the last segment is another long section of polystyrene. It is thermoplastic in nature, with glass transition temperature of the poly butadiene blocks typically -90°C and that of the polystyrene blocks to be +100°C. So, at any temperature between about -90°C and +100°C SBS will act as a physically cross-linked elastomer [42]. It is obtained from local plastic manufacturing company.

Sulfur Modified EVA (Mod-EVA): This polymer is obtained by fusing 50% sulfur powder and 50% Ethylene vinyl Acetate at 150^{o}C, the final product is cooled and shredded into small pallets for further use.

Ethylene vinyl acetate (also known as **EVA**) is the copolymer of ethylene and vinyl acetate. The weight percent vinyl acetate usually varies from 10 to 40%, with the remainder being ethylene. It is a polymer that approaches elastomeric materials in softness and flexibility, yet can be processed like other thermoplastics. The material has good clarity and gloss, barrier properties, low-temperature toughness, stress-crack resistance, hot-melt adhesive water proof properties, and resistance to UV radiation[43].

3.2. Physical Property Tests

3.2.1 ASTM D36: Asphalt softening point test

*Summary of Test Method***:** Two horizontal disks of modified asphalt, cast in shouldered brass rings, are heated at a controlled rate in a liquid bath while each supports a 3.5g steel ball. The softening point is reported as the mean of the temperatures at which the two disks soften enough to allow each ball, enveloped in bitumen, to fall a distance of 25 mm (1.0 in.), as shown in *Figure 3.2*.

3.2.2 ASTM D113: Ductility Test

Summary of test method; The ductility of a bituminous material is measured by the distance to which it will elongate before breaking when two ends of a briquette specimen of the material are pulled apart at a specified speed and at a specified temperature, as shown in *Figure 3.3*. Unless otherwise specified, the test shall be made at a temperature of 25 ± 0.5°C and with a speed of 5 cm/min ± 5.0 %. ETC

Figure 3.2: Softening point test set-up

3.2.3 ASTM D5: Penetration test

Summary of Test Method: The casted asphalt sample is cooled under controlled conditions, then immerse in 25°C water bathe for atleast 90 minutes. The penetration is measured with a penetrometer which releases a standard needle to the surface of the sample under specific load (100g) for 5 seconds. the set up is shown in *Figure 3.5.*

3.2.4 ASTM D4402: Rotational Viscometer Test

Summary of test: RV test measures the torque required to maintain a constant rotational speed of a cylindrical spindle while submerged in an asphalt binder at a constant temperature (135°C). This torque is then converted to a viscosity and displayed automatically by the RV, as can be Seen from *Figure 3.4.*

Figure 3.3: Ductility test samples and set-up

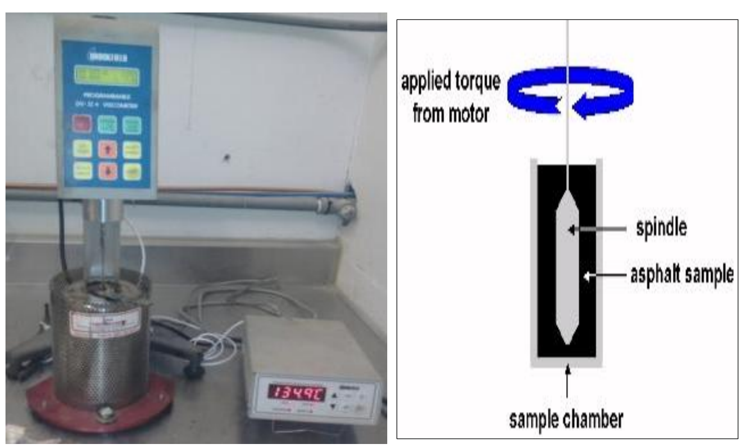

Figure 3.4: Rotational Viscometer (left) and Set-up operating mechanism

3.2.5 ASTM D92: Cleveland open cup Flash point test

Summary of test: the test sample are heated at a constant rate through a controlled heating system, the minimum temperature at which a 0.5 cm flame bulb will ignite the material asphalt evaporating fume (causes it to flash) is measured by the thermocouple thermometer and is recorded as the flash point of that material. Test setup can be seen in *Figure 3.6*.

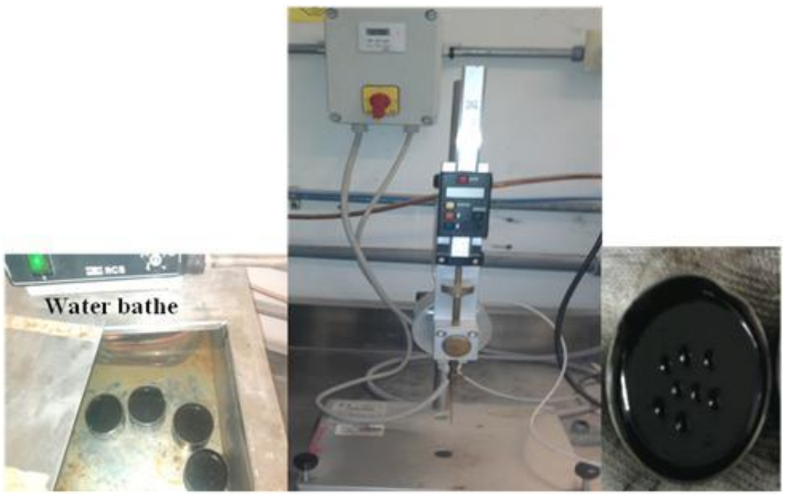

Figure 3.5: Penetration samples in bathe (left), Penetrometer (middle) and Tested sample

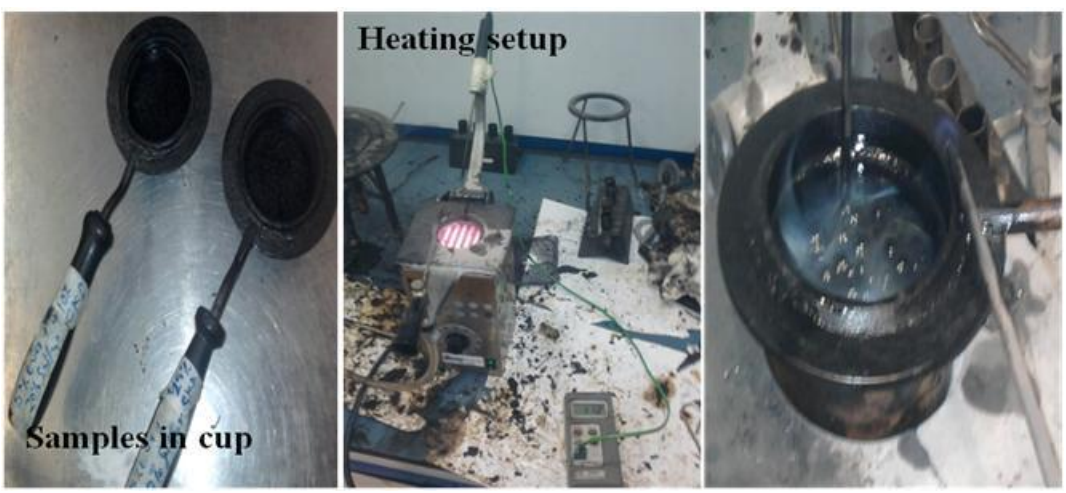

Figure 3.6: Flash point test set-up and samples

3.3 BOND STRENGTH TEST

The bond strength test is devised to measure asphalt mastics ability to resist tensile force and to transfer a sizable amount of force between two bonded surfaces. It is also an indicator of the maximum bond strength the mastic can surmount when subjected to tension.

The test result will be an appropriate parameter that determines the suitability of the mastic in keeping a working/moving-crack relatively intact, which will helps in preventing rapid propagation and widening of such crack. When it also comes to construction or expansion joints, materials having good tensile strength with high elastic recovery will serve better than weak and highly plastic ones that only extend significantly but fails within a small fraction of their extended length. This can be seen at failed sealed joints that opens-up at concrete-sealant interface or within the sealant itself, to give way for water infiltration as shown in *Figure 3.7***.**

Figure 3.7: joint failure modes

Current active specification test method for expansion joint filler (ASTM D545-08: standard test methods for preformed expansion joint filler for concrete construction) has prescribed the means to check and measure the suitability of joint sealant performance and durability through test such as *water absorption test*, *expansion in boiling water test, recovery and compression test*, *extrusion test etc* but failed to include tensile failure test even with the fact that joints opening are bound to widen during winter as they are likely to narrowed in summer season, because the concrete does not only expand but also contract.

Another major common defect exhibited by asphalt water proofing membrane on flat roofing system is the formation of blisters as shown in *Figure 3.8*, which are pockets or cavities containing trapped air but mostly water and keeps expanding with time due to increase in the water or air pressure. Water blistering is the result of prolonged osmotic flow, often over a period of several years. The water is said to be collected within the blister through osmosis diffusion as a result of difference ionic concentration between the temporarily stored rain water on the roof and the one within the blister. Research shows that this defect is mostly associated particularly with cold applied liquid polyurethane polymer modified roofing material as oppose to other similar one like SBS and rubberize membrane, due to its high vapour permeance which makes it susceptible to high osmotic flow rate [37]. The aforementioned research proposed a means of measuring the osmotic flow rate of a given membrane with the view to establishing a limit value for all roofing material so as to serve in developing osmosis-resistant membrane generally.

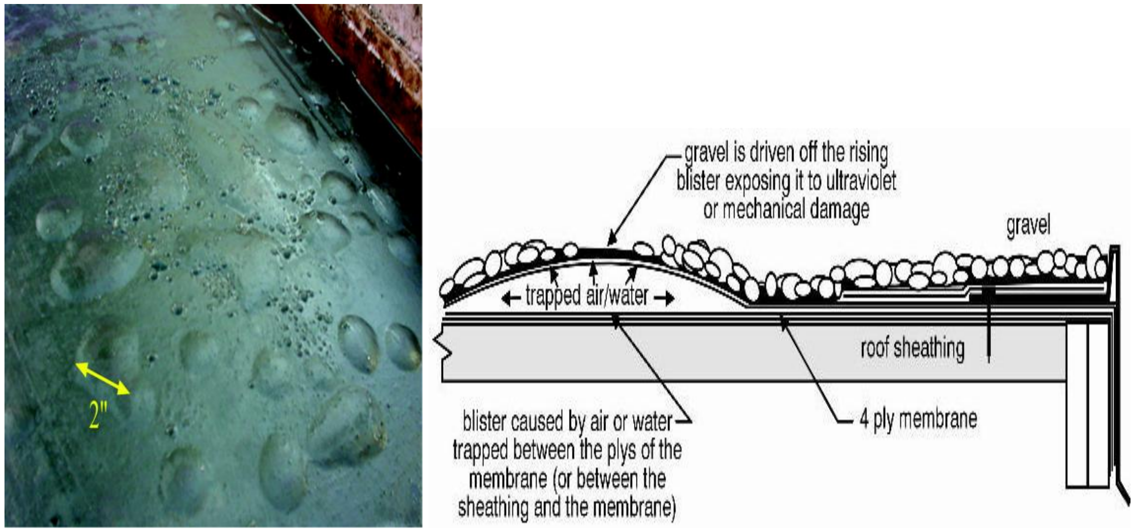

Figure 3.8: Asphalt roofing membrane blisters

Examining the blister formation phenomena critically will help one understand the more many reasons and factors apart from osmosis diffusion that facilitate the whole process. It will only be agreed that osmosis flow of water serve as the genesis of blistering if at all, moisture is the cause of deck-membrane debonding, especially for water blisters. But how can one explain the air blisters? That they are possibly once water blisters but dried with time? Here is a more general reason and explanation; the blisters developed as result of bond loss or insufficient bond strength between the membrane and the roofing deck, period. So, membranes with good tensile strength and having excellent bonding ability will resist blistering better than those deficient in tension but exhibiting the same vapour permeance or osmotic transitivity.

The lack of bonding might be the result in the use of asphalt prime coat that are deficient in tension, or a membrane that has very low tensile strength which make it easier to un-stick, swell and bulge at the slightest given opportunity. During the day when temperature is relatively higher, the asphalt membrane tends to expand generating both shear and tensile strain at the deck-membrane interface. The tensile strain generated is counteracted as a result of lateral/longitudinal movement restriction of the membrane by adjacent wall or other membrane or other part of the membrane that is not receiving the same high sun light energy as depicted by *Figure 3.9*. The same tensile strain is redirected towards the transverse axis of the membrane that is free to move upward, causing a buckling/bulging effects, this in turn creates a tensile force at interface between the membrane and the roofing deck that results in debonding. However this is not the only activity that takes place once there is already a bond failed spot containing trapped air or water. Extra tensile tress comes from increase in air/water pressure contained within the blister space which seems to add to the expansion rate of the blister size.

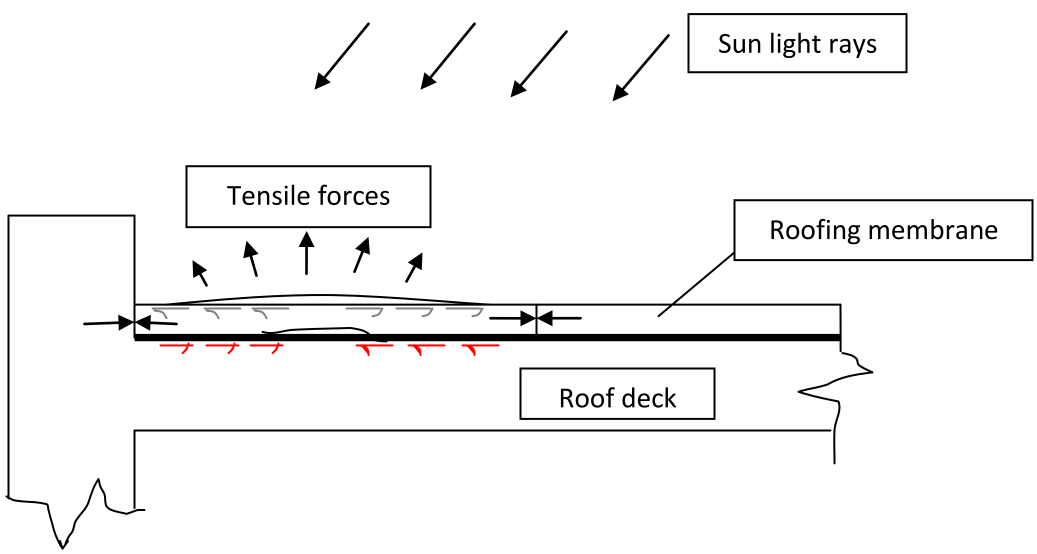

Figure 3.9: Asphalt roofing membrane blistering mechanism

3.3.1 TEST SET-UP AND PROCEDURE

The test involves loading a 30 mm by 20 mm by 6 mm prepared sample of the asphalt mastic in tension at a rate of 1.3 mm/min (1.27 mm/min to be exact) at 25^{o}C, load magnitude and its corresponding deformation measured in the process. The bond strength is reported as the maximum stress recorded which is obtained by dividing the highest load carried by the sample before it fails, by the plate area.

Apparatus: Two 30 mm by 20 mm by 6mm plates, a mechanism which hold and stretch the sample while the load is applied as shown in *Figure 3.10 and 3.11*, a hydraulic or screwed-up device (CBR machine is used in this case and the mechanism converts compression force to tension).

Figure 3.10: Tensile strength test setup and apparatus

The top plate is grooved along its 30 mm sides to just enable it fit in to the sample grip, two holes were also screwed at the center of the 20 mm side of the lower plate for the tightening screws, as shown in *Figure 3.11*.

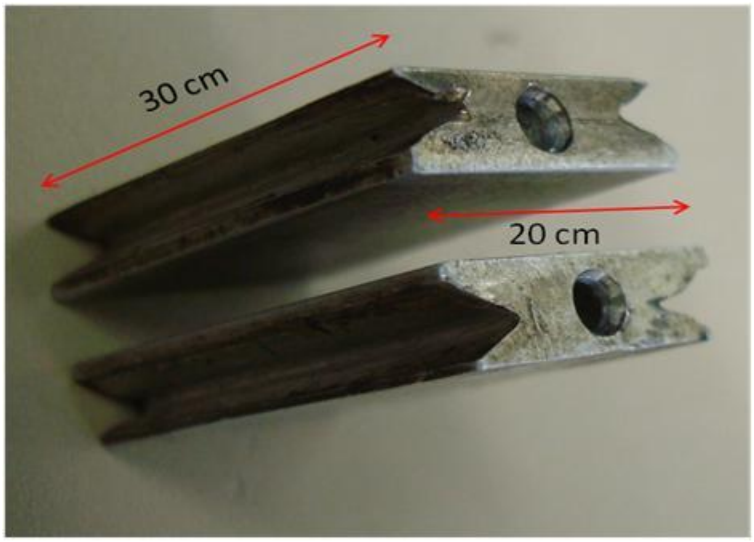

Figure 3.11: Sample plates

The mechanism consist of two parts, the lower main frame which is made-up of two 20 mm thick 75 mm square steel blocks and four 92 mm long cylindrical steel bars which are all assembled to form a stable equi-spaced rectangular frame. A sample grip having a wedged-like edge slot matching the size of the plate is fixed to the upper disk with the aid of a short steel rod that is supplemented with spring bearing to help eliminate any unnecessary compressive force while sample is being inserted. The upper part of the mechanism consist of a 20 mm thick 70 mm circular dist and two cylindrical rods that are attached and spaced in such a manner that they are accommodated and can pass between the main frame arrangement through two bored holes on the upper main frame block. The upper part rested on a bearing or spring suspending system which eliminates any additional load on the tested sample due to the self weight of the upper frame. A screw is transversely inserted to the end of each the two cylindrical rods attached to the upper disk that will fit into the screwed bored holes at the 20 mm long side of the lower sample plate.

Sample preparation: the two plates were spaced 6 mm apart and fixed in position with the aid of a holder, three sides of the arrangement were wrapped with a non-sticking paper. The mastic was heated to a workable state capable of filling the 6 mm x 20 mm x 30mm space without voids and also sticking to plate wall with full strength. The material was allowed to cool sufficiently before unwrapping it, for atleast 15 minutes, when necessary the sample is put in a freezer after 30 minutes for 5 minute to enable the smooth removal of the non-stick paper without sample disturbance. Then the sample was then put in a 25^{o}C water bathe for atleast 90 minutes before testing.

3.4 EXPIREMENTAL DESIGN

The experiment was design to produce asphalt mastics similar to those used in roofing, waterproofing and damp-proofing applications. The filler content ranges between (0-25%) was selected bearing in mind the specified mineral filler content in ASTM D2822: *Asphalt Roof Cement* (45% maximum). Industrially, most of the polymer modified asphalt roofing and waterproofing product contained up to 10% polymer, sometimes even more, hence the polymer range between 0% to10%. Preliminary test shows the oil sludge and sulfur to behave more like extenders rather than fillers above 10% by weight of asphalt content, as they are employed as extenders their composition range selection seemed appropriate.

Table 3.2: Blends experimental design

Asphalt		Heavy oil fly ash 'HOFA'					Cement kiln dust 'CKD'				Lime Dust 'LMD'			
		0%	10%	15%	20%	25%	10%	15%	20%	25%	10%	15%	20%	25%
	0%													
Sulfur	10%													
	20%													
	30%													
Mod-EVA	5%													
	10%													
SBS	5%													
	10%													
Oil Sludge	20%													
	40%													

3.5 SAMPLE PREPARATION AND TESTING

The following sub-headings contains the detailed description of how the various mastic were prepared before testing. Different oil bathe mixing temperature and mixing duration were adopted for the diverse mastic composition in accordance with previous work and the properties of the additives involved.

Neat asphalt was put into an oven at 140^oC to 150^oC for a sufficient period of time to obtain a liquid binder that can be easily poured. The additive fillers are also placed in the oven for 24 hours at 100^oC – 105^oC prior to mixing in order to eliminate absorbed moisture.

3.5.1 Polymer-filler mastics

800g of the liquid asphalt was placed in the 1000 ml mixing container, which was in turn fixed in a 140^oC oil bathe-mixer setup as shown in Figure 3.12, and stirred continuously for sufficient time to establish thermal equilibrium. Then a measured quantity of modified-Eva (5% or 10%) by weight of the binder was introduced, and mixing continued at a speed of 1500 rpm for about 10 minutes. After which 200g of the mix was placed in four fresh cans and stored temporarily (less than 30 minutes) for subsequent blending. The various 200g polymer-asphalt blends were further combined with 10%, 15%, 20% and 25% of Oil fly-ash, Cement kiln dust or Lime dust by weight of the polymer-asphalt mix for 5 minutes. Samples were casted immediately for each blend after final mixing.

The above paragraph described the mixing procedure of sulfur-modified Eva with filler additives. The process of mixing SBS polymer with filler within asphalt differs in terms of manner and duration of mixing. Finely grounded SBS powder having high specific surface area was employed, 420g of liquid asphalt was mixed with 5% or 10% of the SBS at 130-140^{o}C, the temperature of this mix was then raise to around 160^{o}C while stirring manually with a spatula spoon to ensure uniform distribution of the polymer particles. The can was then sealed with the aid of Aluminum foil and paper tape, and place inside the oven for approximately 12 hour at 130^{o}C for the SBS particulate to swell and soften. This mix was then place in oil bathe at 190-200^{o}C and sheared with a high speed mixer (1500 rpm) for 20 minutes. Resulting blend was further divided into two 200g new container for final mixing with the mineral fillers. The mineral addition is also done at this same temperature and speed for 5 minute. The sealed storing helps eliminate the necessity of a long duration mixing which also reduced the effect of asphalt oxidation.

Figure 3.12: Mixer-Oil bathe setup

3.5.2 Polymer-sulfur-filler mastics

800g of the liquid asphalt was placed in the 1000 ml mixing container, which was in turn fixed in the 140^o oil bathe-mixer setup and stirred continuously at about 1500 rpm for sufficient time to establish thermal equilibrium. Then a measured quantity of modified-Eva (5%) by weight of the binder was introduced, and mixing continued for about 10 minutes. Additional sulfur additive (20 or 30%) by weight of asphalt was then further introduced in the mix, and the stirring continues for another 10 minutes. After which 200g of the mix was placed in four fresh cans and stored temporarily (less than 30 minutes) for subsequent blending. The various 200g polymer-asphalt mix were further combined with 10%, 15%, 20% and 25% of Oil fly-ash, Cement kiln dust or Lime dust by weight of the polymer-asphalt mastic for 5 minutes. Samples were casted immediately for each blend after final mixing.

As for the SBS-sulfur-filler mixes, finely grounded SBS powder was employed, 420g of liquid asphalt was mixed with 5% of the SBS at 130-140^{o}C, the temperature of this mix was then raised to around 160^{o}C while stirring manually with a spatula spoon to ensure uniform distribution of the polymer particles. The can was then sealed with the aid of Aluminum foil and paper tape, and place inside the oven for approximately 12 hour at130^{o}C for the SBS particulate to swell and soften. This mixture was then place in oil bathe at 190-200^{o}C and sheared with a high speed mixer (1500 rpm) for 20 minutes. Resulting blend was further divided into two 200g new container for final mixing with the mineral fillers. The fillers addition was also done at this same temperature (200^{o}C) and speed for 5 minute. Finally, the appropriate amount of sulfur was added and the mixing carried on for 2 minutes maximum. The mix is then put in to an oven for atleast

20 minutes at temperature above 170^{o}C for the vulcanization process to take complete form before test samples were casted.

3.5.3 Sulfur and Sludge-filler mastic

800g of neat asphalt was poured into a 1000 ml mixing can, the can was then placed in an oil bathe at 140^{o}C – 145^{o}C, the asphalt is continuously stirred with the aid of a high speed shear mixer until temperature equilibrium between the bathe and the container has been established. The above temperature has been selected due to the fact that beyond 145^{o}C the sulfur extender has more tendency to liberate in form of gaseous compound that are toxic. Appropriate amount of sulfur (10%, 20%, & 30%) by weight of the 800g asphalt was introduced in to the mix, and the stirring continued for 10 minutes. Afterwards 200g of the final mix was poured into four new mixing can and stored temporarily for not more than 30 minutes inside oven at 145^{o}C. The four blends were then mixed in the same manner as previously mentioned with appropriate filler content (10%, 15%, 20%, 25%) but for 5 minutes, test samples were casted immediately for each blend to avoid a prolong storage additive settlement or separation.

All the process described for the preparation of sulfur-filler mastics including timing, filler composition, temperature of oil bathe and mixing holds true for sludge filler blends.

3.6 STATISTICAL ANALYSIS AND REGRESSION MODELLING

The data generated from the previous physical tests were subjected to statistical scrutiny via analysis of variance (ANOVA) by a Minitab (version 16) software [40]. Due to the nature of the experimental design and the number of treatment involve in each test, two-way ANOVA was selected for the task. But before ANOVA was selected for the analysis, model adequacy check was performed on most of the data. Test such as 'Equality of variance test' and 'Normality check test' were carried out to ensure the relevance and appropriateness of the selected test method. The observations were independent and not influence by one another, so independency assumption was satisfied, the normality and equal variance checks also shows the data to be normally distributed with more or less equal variance for each level of treatment. For the few cases were the data does not follow the normal distribution, the deviation seem to be slight and can be accommodated by the analysis method (ANOVA) due to the sufficient sample size. For more detailed insight on the analysis of variance see *appendix C*.

Regression models were then generated for the properties tested with the same Minitab-16, the properties were modeled as dependent variables with the treatment (additives) as the independent variables. Before any model is generated, the actual response surface was plotted and observed, this gives the general idea of the suitable function variables that will enable the smooth fitting of the model to the actual response surface. For instance a linear plane response surface means no quadratic terms are required for accurate depiction of the model plane, otherwise curving of the response plane on the side of one or both additive will prompt the inclusion of quadratic term of that additive or all additives, it could also be cubic term. After assessing the possible suitable function for a model, potential suitable models were generated, first with only the treatments (say X1 and X2) as variables. Then all other possible suitable combination were generated, which include quadratic and or cubic terms depending on the shape of the actual response plane. Comparisons were made, and the best model to represent that property change was selected based on the adjusted correlation coefficient value ($R^2_{(adj)}$) of the model, the significance of terms in the model equation and other factors such predicted coefficient of correlation ($R^2_{(pred)}$).

CHAPTER 4

RESULTS AND DISCUSSION

The results obtained were discussed for each test separately. Starting with the softening point, followed by the ductility test, then the penetration test results were examined, the viscosity was discussed before the Flash point test results, and finally the bond strength test result was analyzed.

4.1 Softening Point (SP)

Softening point results of the various asphalt mastics were studied in sequence according to group containing polymer, sludge, sulfur or combination of sulfur with polymer. Each of the above mastic is considered for all filler (HOFA. CKD and LMD) composition as one group. The polymer mastics were first analyzed, followed by the polymer-sulfur mastics, then the sludge mastic results was studied before the results from sulfur blends were discussed.

4.1.1 Modified Eva-Filler mastics

The softening point 'SP' tend to rise uniformly with increase in mod-EVA content as expected, an average of $2^{o}C$ increment is recorded for every 5% addition of mod-EVA as shown in *Figure 4-01*. The resulting SP of HOFA-mod-Eva blends (double additive) is generally less than that of a pure HOFA for the 5% mod-EVA curve. This might be due to the plastomeric nature of the mod-EVA and its sulfur content, which makes it (mod-EVA) presence in large proportion necessary for the formation of more stable HOFA-mod-EVA composite. But the 10% mod-EVA curve exhibit almost the same SP values as the 0% mod-EVA graph, this supports the assertion made earlier of having much harder material with more mod-EVA for the two-additive mastics true.

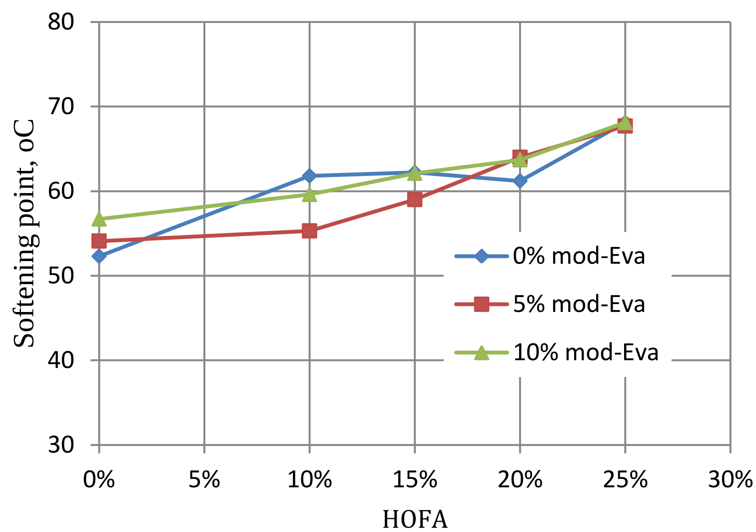

Figure 4-01: Softening point of HOFA-Mod-EVA mastic

As for LMD-mod-EVA blends, for LMD 20% content and below, the SP do not seem to be affected, which is also the case for the LMD-only blends, see *Figure 4-02*. But the 10% mod-EVA curves shows slightly higher SP than the 0% mod-EVA graph, which implies that obtaining an LMD-mod-EVA material with higher SP than the LMD only mastics is only possible if the mod-EVA content equals or exceeds 10%.

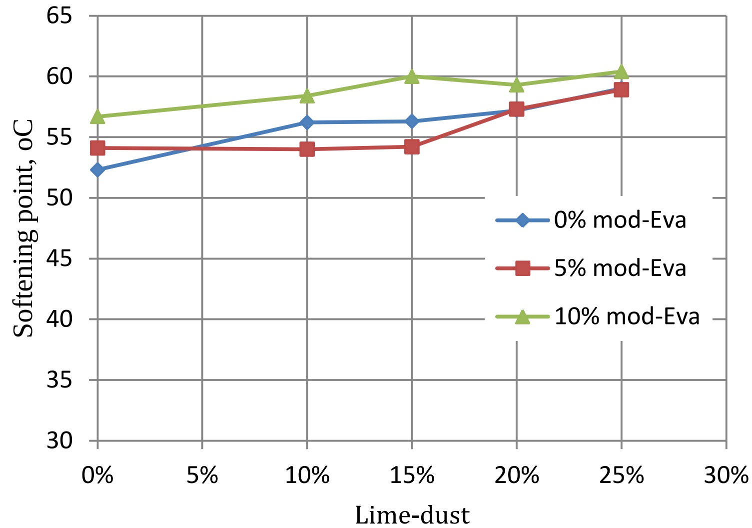

Figure 4-02: Softening point of LMD-Mod-EVA mastic

The CKD has little influence on the SP for the mod-EVA asphalt mixture. But the two additives blends demonstrates higher SP than the CKD only blends even for 5% mod-EVA mastics, as can be seen from *Figure 4-03*, unlike the LMD case.

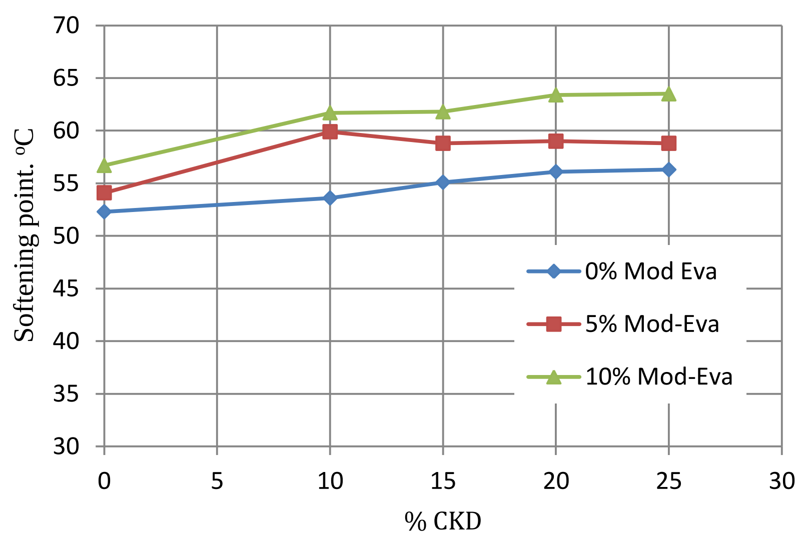

Figure 4-03: Softening point of CKD-Mod-EVA mastic

4.1.2 Styrene Butadiene Styrene-Filler mastics

Styrene Butadiene Styrene 'SBS' has a tremendous effect on the SP of the asphalt. The initial 5% SBS content yield material with an SP increase of about 34^{o}C compared to the neat asphalt, as it is shown by *Figure 4-04***.** This might be due to the high melting point of the SBS polymer (180^{o}C) which when dispersed between the relatively soft asphalt matrix produce composite having the asphalt molecule physically reinforced within the polymer chain/network. The SP results are obtained using glycerol as the fluid heating medium for the SBS-only blends.

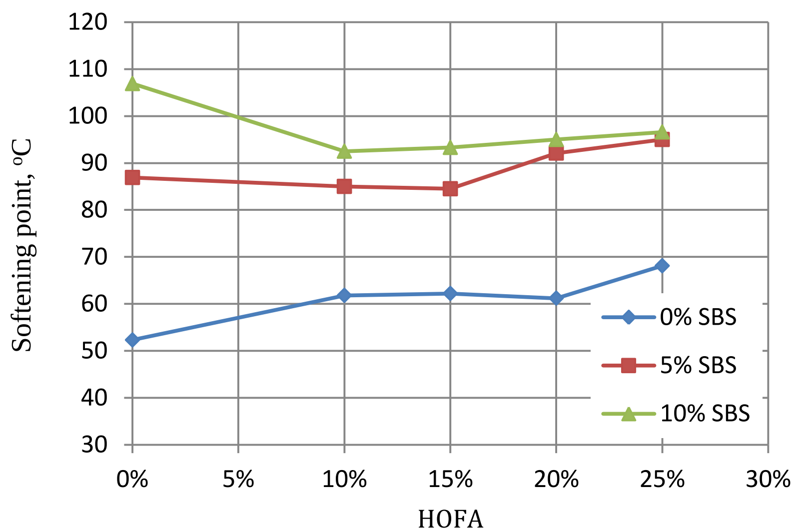

Figure 4-04: Softening point of HOFA-SBS mastic

Adding fillers (HOFA, LMD & CKD) to the SBS-modified asphalt results in materials with fairly less SP value than the SBS-only blended asphalt material at lower doses of the fillers while materials with even higher SP than the SBS-only mastics are obtained at higher dosage (20-25%) of these fillers, *Figure 4-04, 4-05 & 4-06* shows evidence of the above claim. This is because adding the filler in large quantity will tend to raise the required amount of energy needed to soften the materials by forming a closely parked structure of the filler-SBS and asphalt matrix. But depending on the amount of SBS polymer presence, lower content of filler will only result in weakening the asphalt-SBS structure due to loss of adhesion, which cannot be compensated by the little increase of specific heat energy from introduction of the filler.

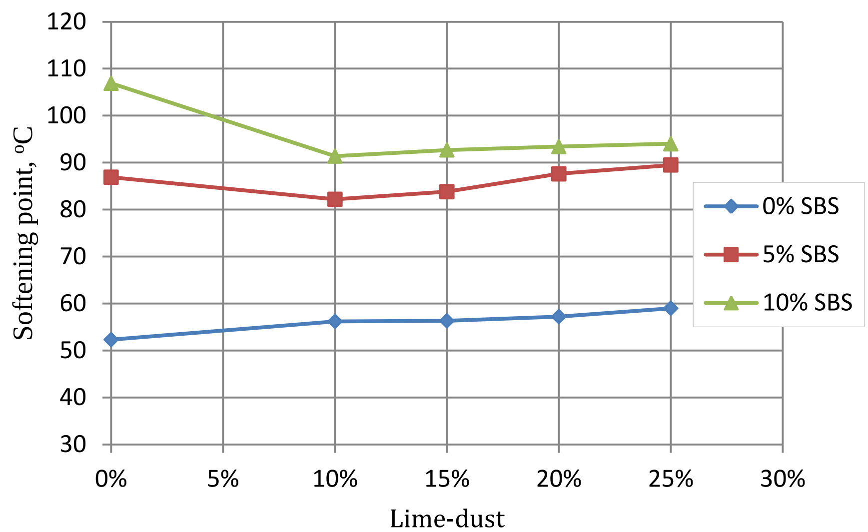

Figure 4-05: Softening point of LMD-SBS mastic

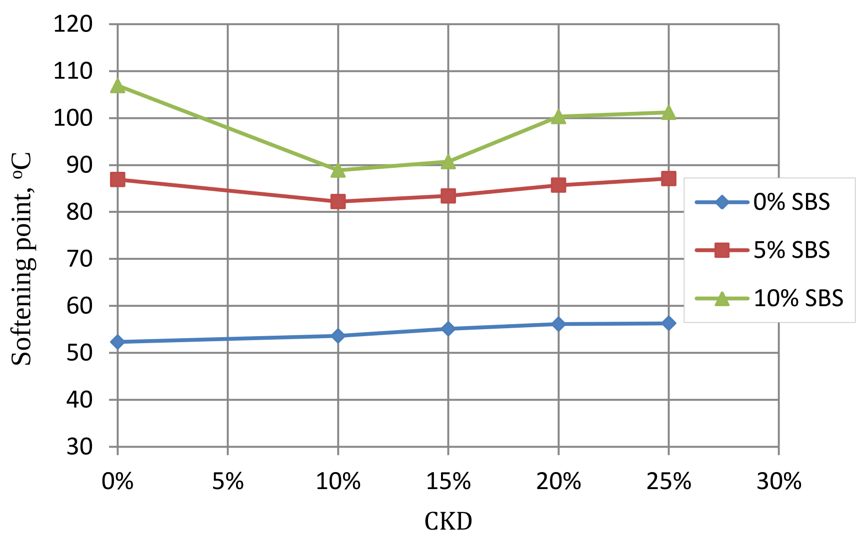

Figure 4-06: Softening point of CKD-SBS mastic

4.1.3 Mod-EVA, Sulfur, Filler – mastics

It has been previously shown that the addition of LMD mineral filler to 5% Mod-EVA-asphalt has little effect on the softening point (SP), especially at lower content (0 – 15 %) range. So were the case with 20% and 30% sulfur containing 5% Mod.EVA asphalt blends. The 20% and 30% sulfur blends tend to have almost the same SP value for equal LMD content, based on trend from *Figure 4-07*. The same applies to CKD mineral filler, see *Figure 4-08.*

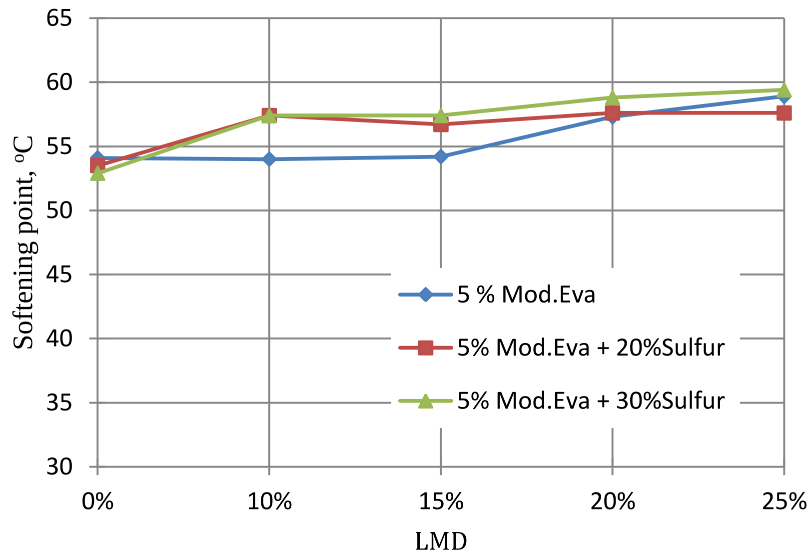

Figure 4-07: Softening point of LMD-Mod-EVA Sulfur mastic

The HOFA however have a relatively significant influence on the SP within both the 5% Mod-EVA-only and 5% Mod-EVA-Sulfur blends compared to CKD and LMD fillers. A gradual increase in SP with more HOFA filler can be observed from *Figure 4-09*, but the 20% and 30% sulfur blends exhibit only a little difference in SP.

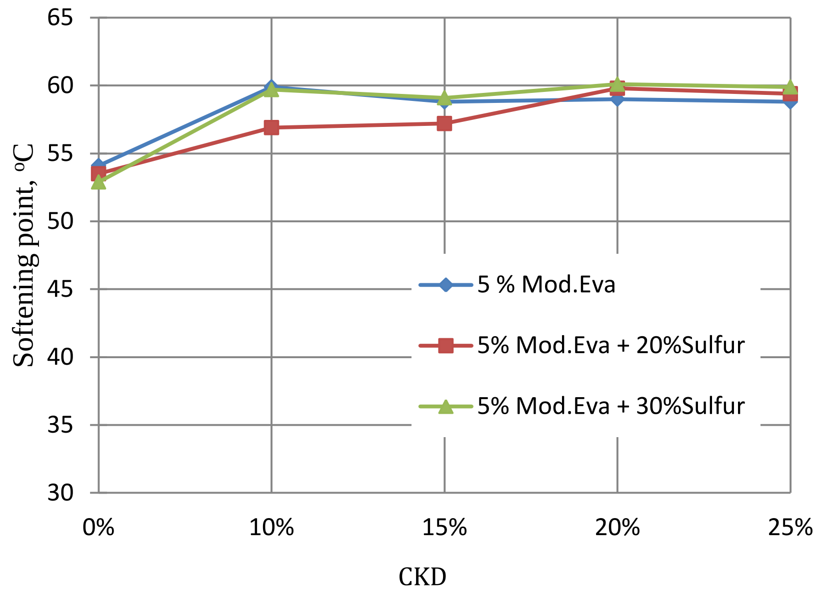

Figure 4-08: Softening point of CKD-Mod-EVA Sulfur mastic

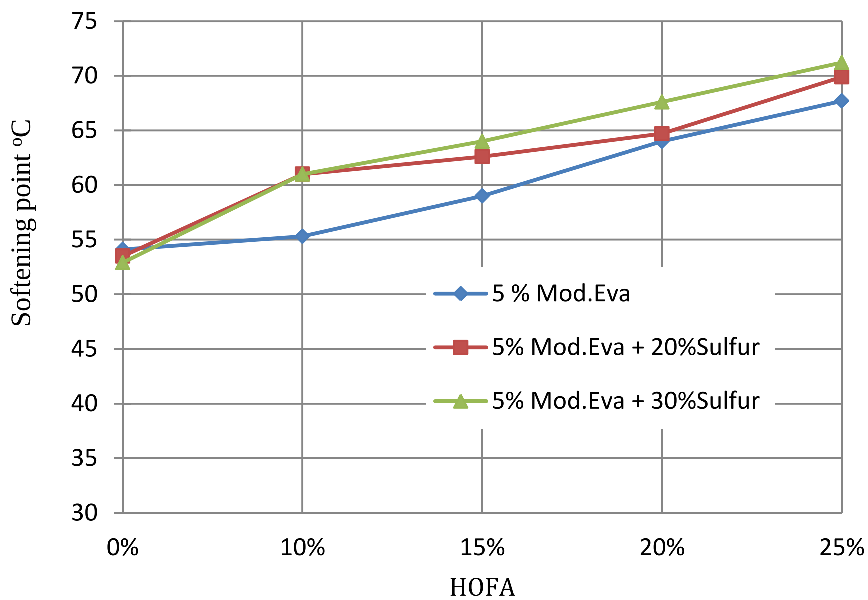

Figure 4-09: Softening point of HOFA-Mod-EVA Sulfur mastic

4.1.4 Sludge – Filler mastics

The softening point (SP) drops with increase in the amount of oil-sludge (OS) for doses above 10% by weight of the asphalt, below this quantity it seems to be rising just like in the case with sulfur as seen from *Figure 4-10*.

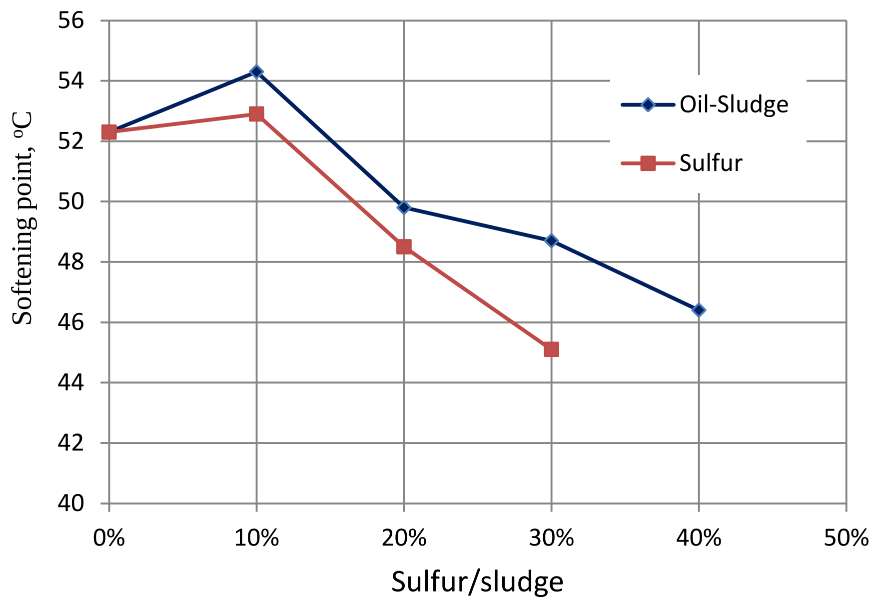

Figure 4-10: Softening point of Sulfur & Sludge mastic

The above behavior might be the result of the kind of role played by these additives, that is, whether as hardening or softening agents depending on the proportion of asphalt replaced, but could also be due to the fact that, the actual asphalt matrix remain dominant and keep strengthening with more of these additive below this range (10% by weight of asphalt), after which a reversal in property effect is observed. So the sludge act as filler at quantities below 10% by weight of asphalt and behaves as flux above this value.

The HOFA usually raises the SP-value appreciably but becomes less effective when combined with sludge at lower HOFA (15% and below), as can be seen from *Figure 4-*

11. However there happens to be constructive interaction between the two substance at higher content of HOFA (20% and above), which yield an SP-value that are higher than those of HOFA-only blends. Each addition of 20% oil sludge causes a decrease of about 3^{o}C in SP-value for sludge-only blend, but combining 25% HOFA with 40% oil sludge will yield material composite with about 60% higher SP than the neat asphalt.

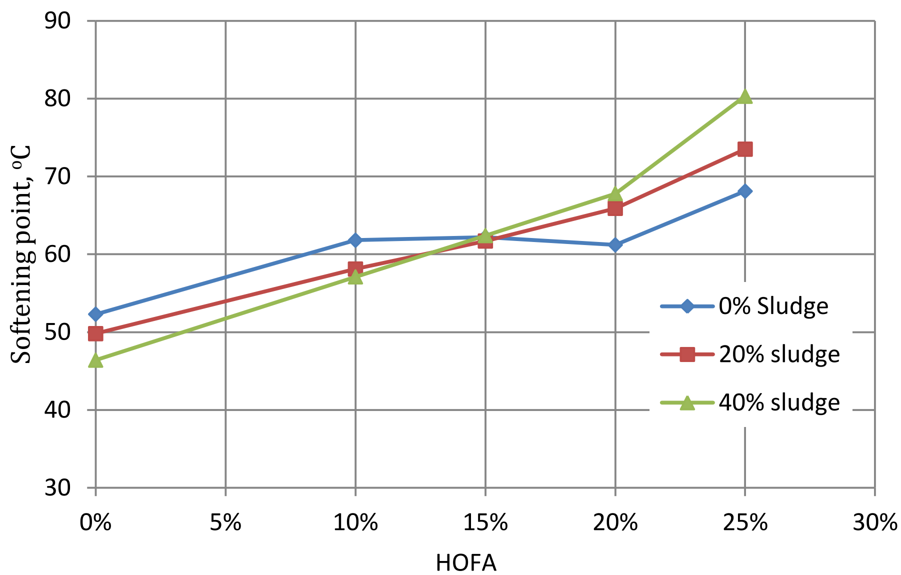

Figure 4-11: Softening point of HOFA-Sludge mastic

The CKD and Oil sludge shows similar constructive interaction when combined within the asphalt medium with a positive effect on the SP-value, but not as significant as in the case of HOFA with OS blends. *Figure 4-12 shows that* there is just 7^{o}C increase in SP for 25% CKD and 40% OS blend compared to pure asphalt.

There seem to be little or no interaction between the LMD and OS for the given content ranges. Lower SP values are observed for higher OS-content-blends containing the same amount of LMD than blends having no OS, as seen from *Figure 4-13*. 25% LMD and 40% OS blend have lower SP relative to the neat asphalt.

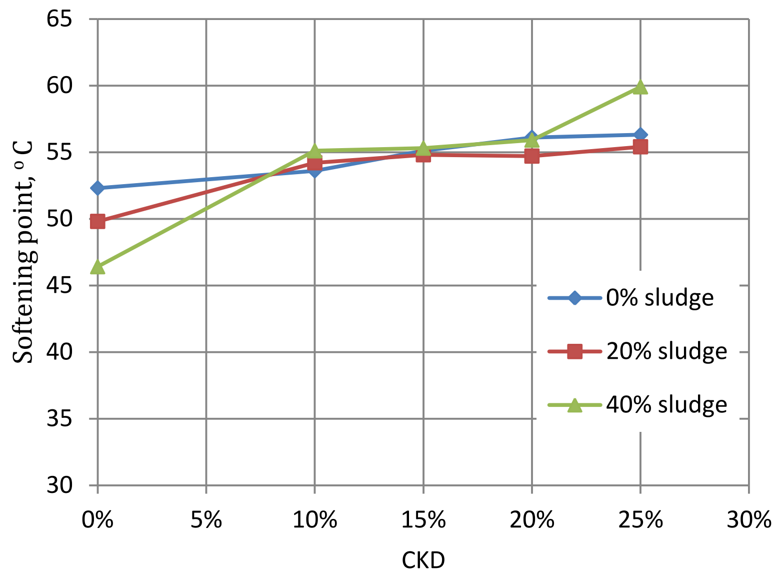

Figure 4-12: Softening point of CKD-Sludge mastic

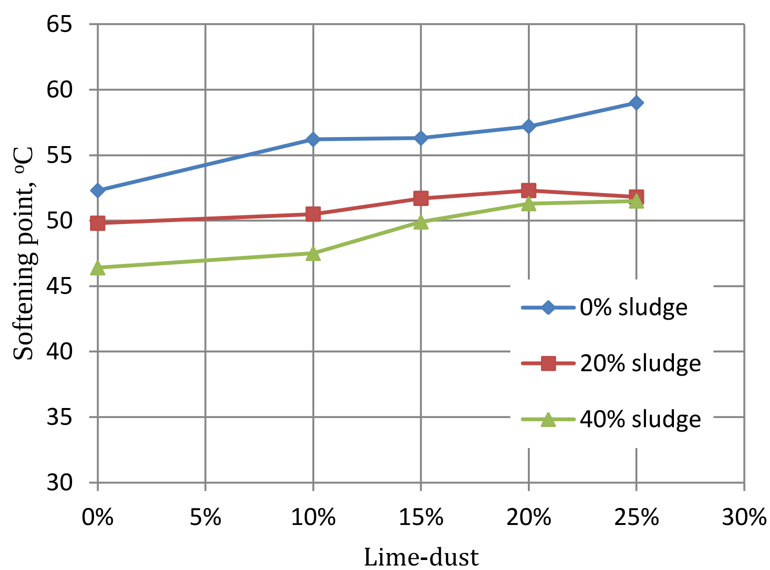

Figure 4-13: Softening point of LMD-Sludge mastic

4.1.5 Sulfur-filler mastics

Generally the softening point is negatively affected with more sulfur additive and positively influenced with increasing HOFA, as shown by *Figure 4-14*.

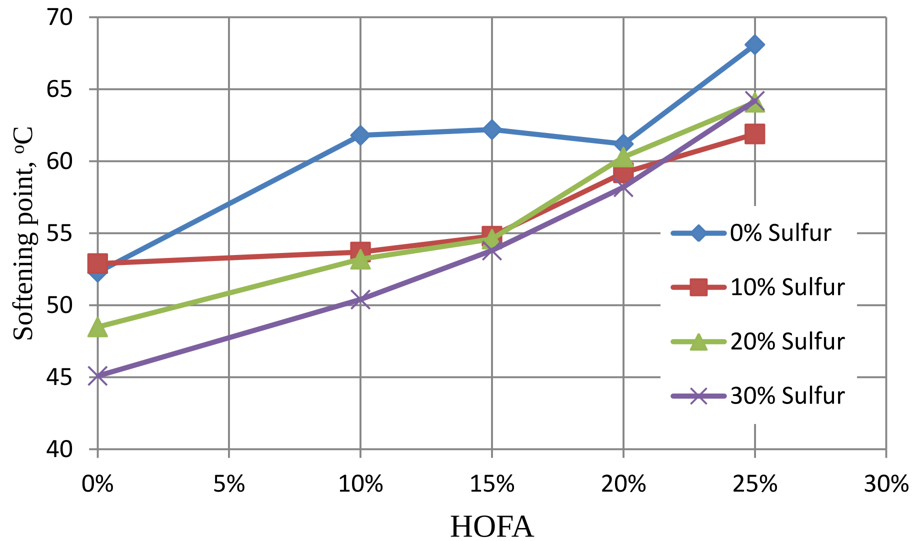

Figure 4-14: Softening point HOFA-Sulfur mastic

In pure HOFA-asphalt blends, an insignificant change can be observed within 10 – 20% HOFA content, but a drastic additional rise by about 7°C in softening point was recorded for blend containing 25% HOFA. The initial 10% HOFA has already resulted to almost 18% rise. On the other hand, addition of sulfur to the neat bitumen does not seem to change the SP within 0 – 10% range, but at higher doses (20 – 30%) the SP seems to diminish by approximately 13%. Combining both sulfur and HOFA seem to have an overall destructive resultant, sulfur will lower the SP while HOFA will tend to push it up, an equilibrium value that is in-between pure Sulfur/HOFA blend will always be the resulting SP, depending on relative proportion of these additives, but will be closer to the HOFA-asphalt blend value for equal weight combination.

An increase in softening point is observed with higher lime dust (LMD) content, from *Figure 4-15*, but unlike HOFA, the LMD has little influence on this parameter. A 7% rise is recorded for the initial 10% composition, and 25% was able to annul the dwindling effect of 30% sulfur on the SP.

In contrast to HOFA and LMD, the effect of CKD on softening point can be observed from *Figure 4-16* to be uniform for CKD-only blends. An average increment of 1^{o}C for every 5% additional CKD contents can be seen. 15% was enough to nullify the lessening effect of sulfur on the softening point. But 10% sulfur curve proves to be the softest combination, which might be due to relatively lesser amount of filler grains (sulfur and CKD combined).

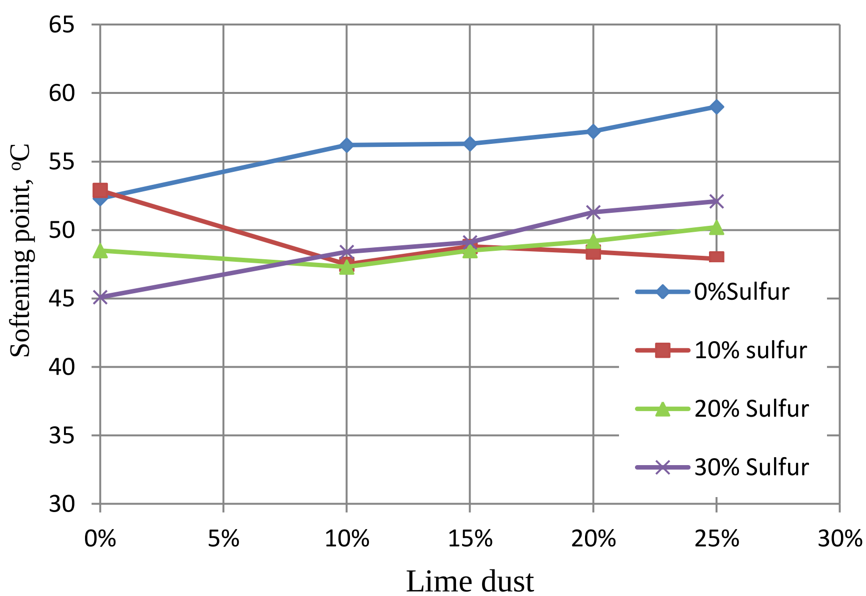

Figure 4-15: Softening point of LMD-Sulfur mastic

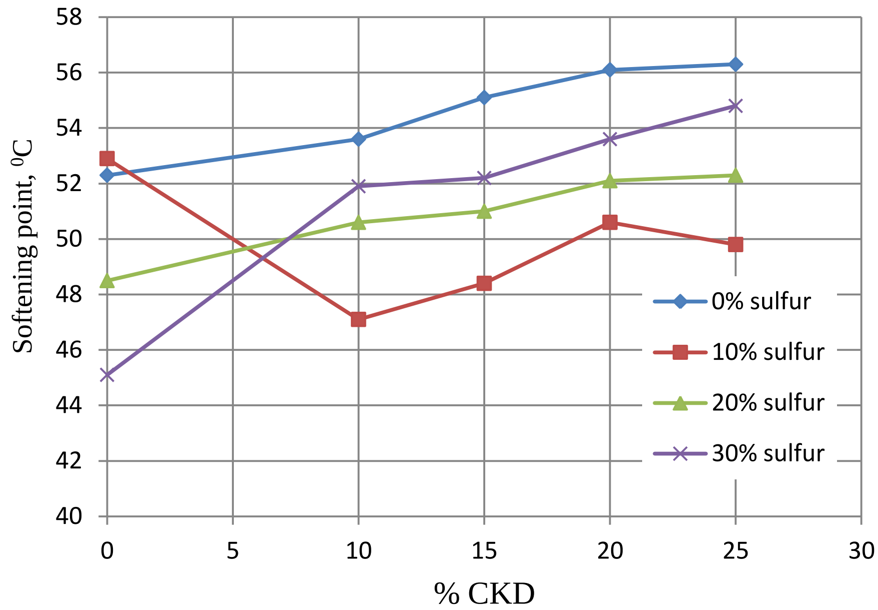

Figure 4-16: Softening point of CKD-Sulfur mastic

4.2.0 DUCTILITY

Ductility test results of the various asphalt mastics were studied in sequence according to group containing polymer, sludge, sulfur or combination of sulfur with polymer. Each of the above mastic is considered for all filler (HOFA. CKD and LMD) composition as one group. The polymer mastics were first analyzed, followed by the polymer-sulfur mastics, then the sulfur mastic results was studied before the results from sludge blends were discussed.

4.2.1 Modified Eva-Filler mastics

Modified-EVA 'mod-EVA' results in material blend with lower ductility, 5% of Mod-Eva yield composite having 50% less ductile property relative to the original asphalt, and the 10% content causes about 75% loss in ductility as seen from *Figure 4-17*. It is also obvious that this trend might continue with more mod-EVA. But the two additive blends containing both HOFA and mod-EVA (5% curve) tend to have fairly higher ductility than the HOFA-only blends. This can also be attributed to the plasticizing nature of the EVA component, and also the presence of sulfur in the mod-EVA. On the other hand, further addition of the mod-EVA (10% curve) to the two-additive blend results in less ductile material than the HOFA-only.

There seem to be continuous decline in the ductility with both the addition of LMD and mod-EVA. And the double-additive blends generally results in material having lower ductility than the LMD-only or mod-EVA only blends as results from *Figure 4-18* implied.

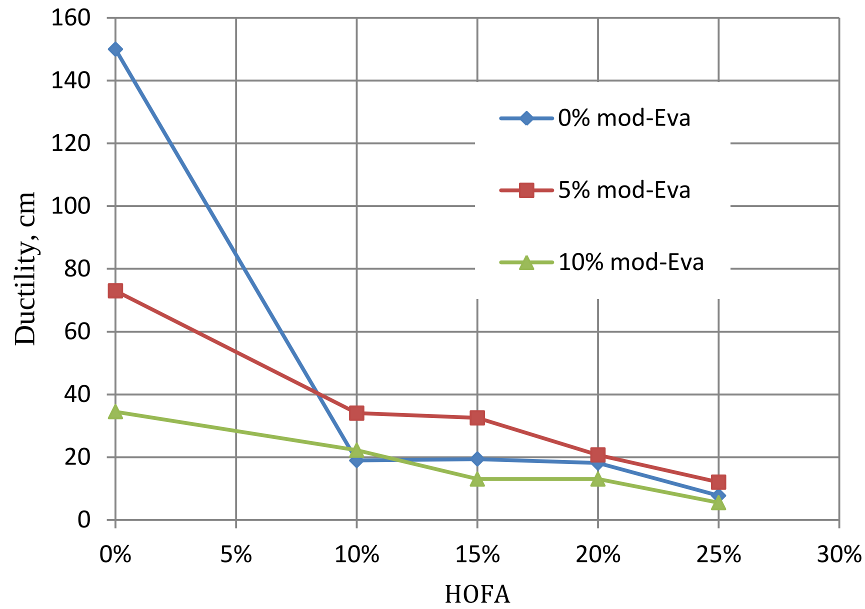

Figure 4-17: Ductility of HOFA-Mod-Eva mastic

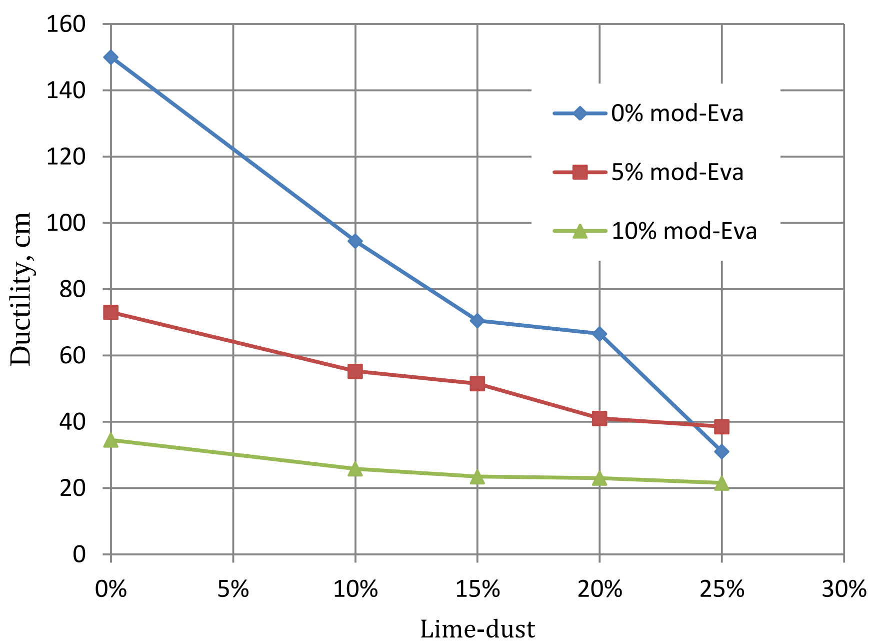

Figure 4-18: Ductility of LMD-Mod-Eva mastic

The pattern and trend as in the LMD-mod-EVA blends can be observed with CKD-mod-EVA ones, from *Figure 4-19*. The only difference here is that the CKD has much higher decreasing effect on the ductility than LMD, so the resulting CKD-mod-EVA composite mostly has lower ductility than their LMD counter parts.

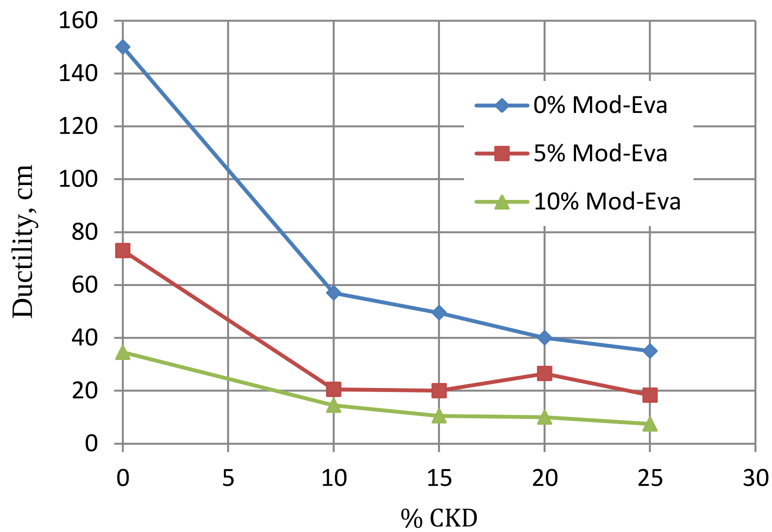

Figure 4-19: Ductility of CKD-Mod-Eva mastic

4.2.2 Styrene Butadiene Styrene-Filler mastics

Less ductile materials results from the addition of Styrene Butadiene Styrene 'SBS' to the asphalt. 10% SBS is sufficient to almost eliminate the ductile property of the original asphalt completely. And the addition of HOFA filler causes further decline in this property for both the 5% and the 10% SBS containing mastics, as *Figure 4-20* shows**.**

Similar behavior as with HOFA is observed with CKD filler from *Figure 4-21*, but for the LMD blends, the ductility tend to rise relative to 0% filler blends at lower filler content for the 5% SBS curve before it declines, as *Figure 4-22* shows. This could be attributed to the LMD's ability/property to cause a uniform ductility decreasing rate unlike the other fillers, as can be seen from the 0% SBS graph.

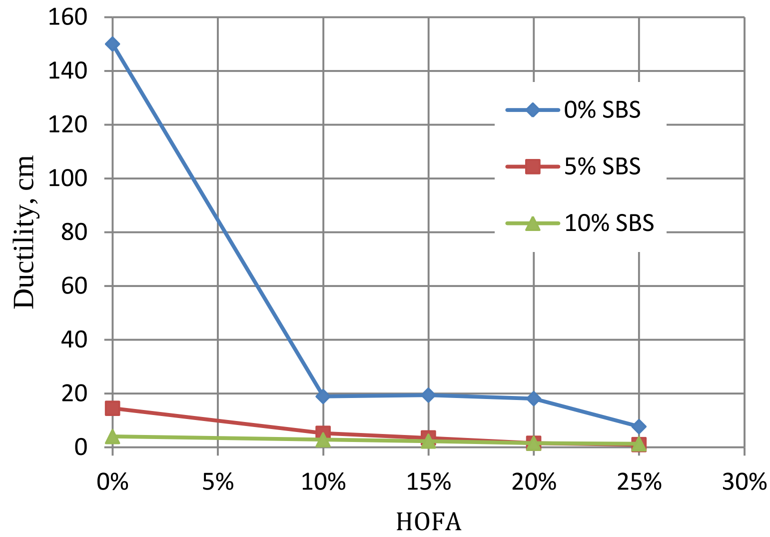

Figure 4-20: Ductility of HOFA-SBS mastic

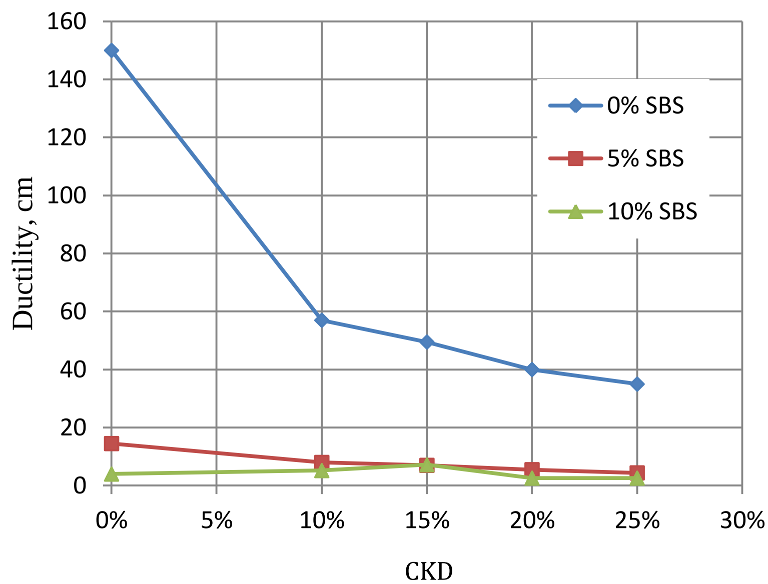

Figure 4-21: Ductility of CKD-SBS mastic

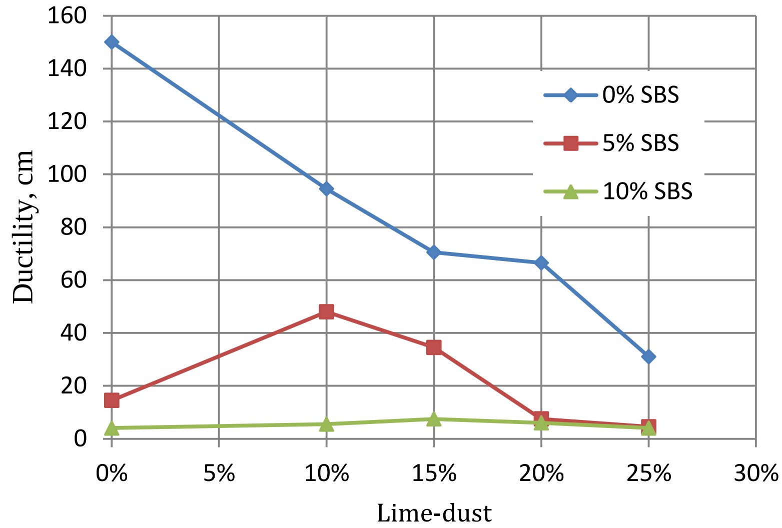

Figure 4-22: Ductility of LMD-SBS mastic

4.2.3 Mod.EVA, Sulfur, Filler mastics

Even though the ductility decreases gradually with increasing LMD content for the 5% Mod-Eva blend, the 20% and 30% sulfur containing 5% Mod-Eva demonstrate almost exactly the same ductility throughout the 10 – 25% filler content range, *Figure 4-23*.

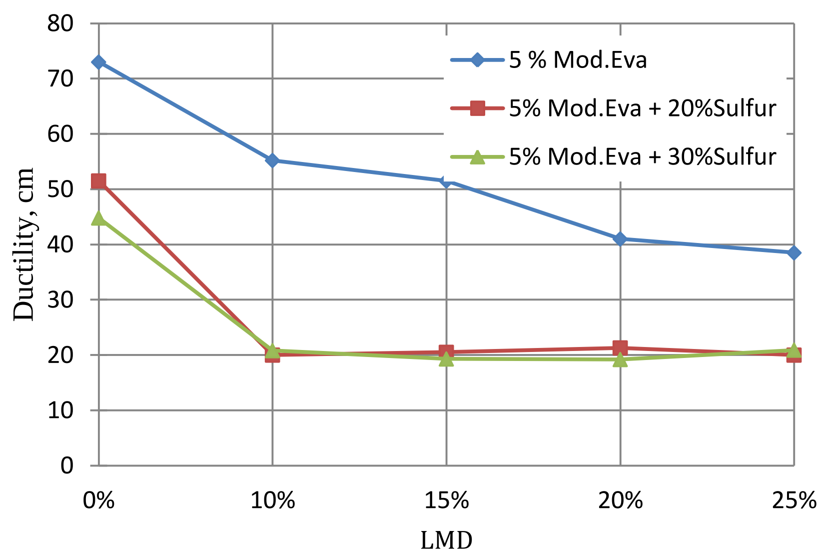

Figure 4-23: Ductility of LMD-Mod-Eva Sulfur mastic

Same trend as above applies to CKD, but here even the 5% Mod-Eva-only blend shows no obvious change in ductility with higher CKD, while the blends containing CKD have a much lower ductility than those without, as can be seen from *Figure 4-24*.

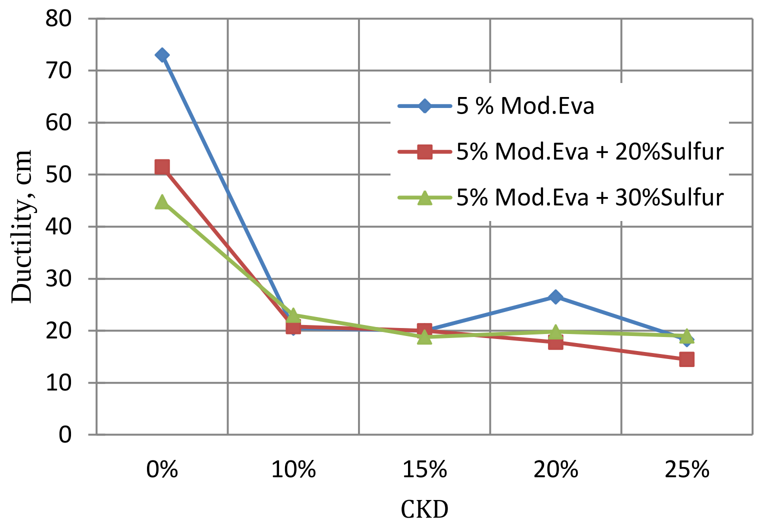

Figure 4-24: Ductility of CKD-Mod-Eva Sulfur mastic

As can be observed, the negative impact of both HOFA and sulfur tend to be maintained within the Mod-Eva modified asphalt, refer to *Figure 4-25 and Figure 4-26*.

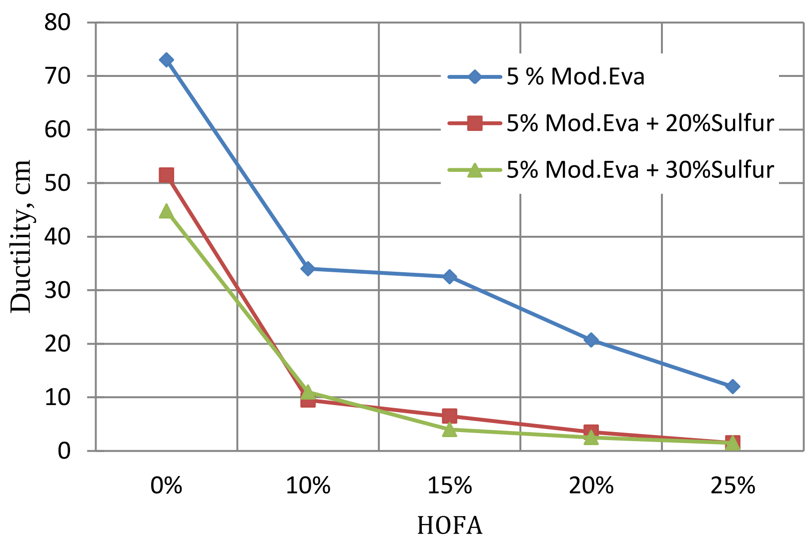

Figure 4-25: Ductility of HOFA-Mod-Eva Sulfur mastic

4.2.4 Sulfur – Filler blends

Addition of sulfur to the asphalt results in low ductile composite, a loss of more than 50% in ductility can be observed at 20% sulfur content from *Figure 4-26*.

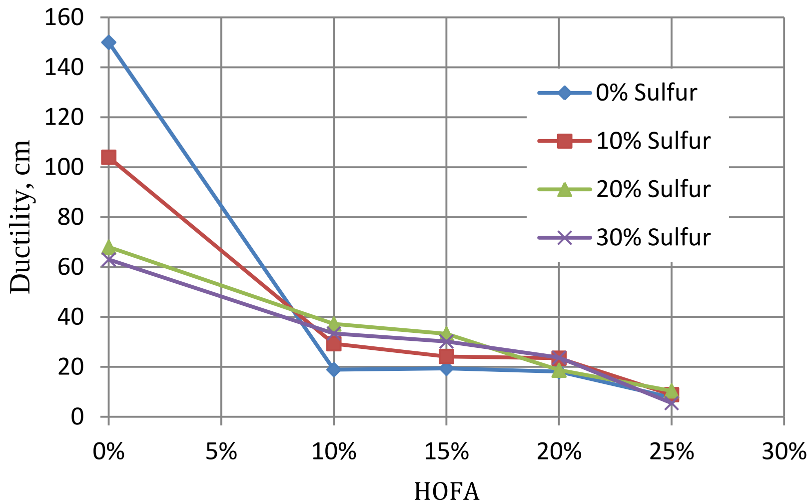

Figure 4-26: Ductility HOFA-Sulfur mastic

More significant reduction is evident with the addition of HOFA to the neat asphalt, initial 10% has eliminates more than 85% of the fresh asphalt ductile property. Even though both sulfur and HOFA negatively affect the ductility individually, a material with relatively higher ductility than purely HOFA-blend is obtained when they are combined.

The ductility also decreases with more lime dust, but not as considerably as in the case of HOFA. A drop of about 35% can be seen at 10% LMD content as shown in *Figure 4-27*. When combined together with sulfur, the resultant ductility always seemed to be lower than both result obtained with individual additives used alone.

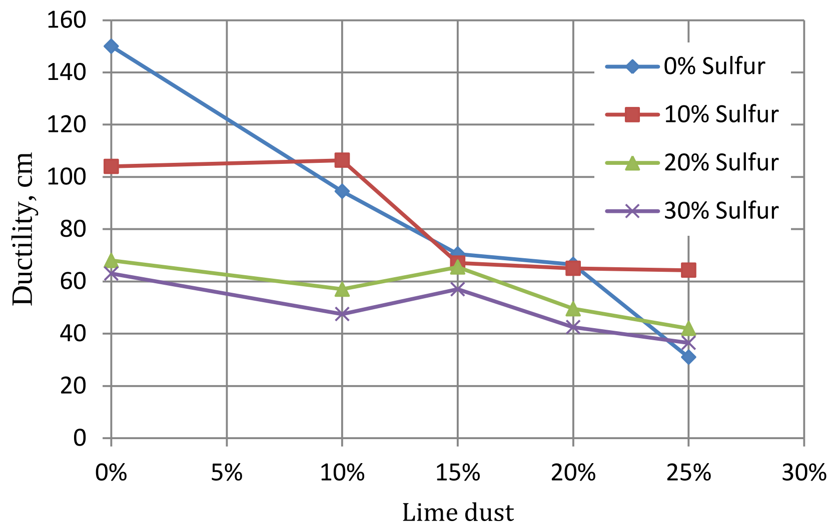

Figure 4-27: Ductility of LMD-Sulfur mastic

CKD-asphalt blend shows lower ductile behavior compared to their LMD blend counter parts, a 67% loss in ductility is recorded as compared to 35% for lime dust – asphalt blend. But as in sulfur–lime blend, the resulting ductility of combined sulfur-CKD is lower than that of either CKD-alone or sulfur-only blend, as seen from *Figure 4-28*.

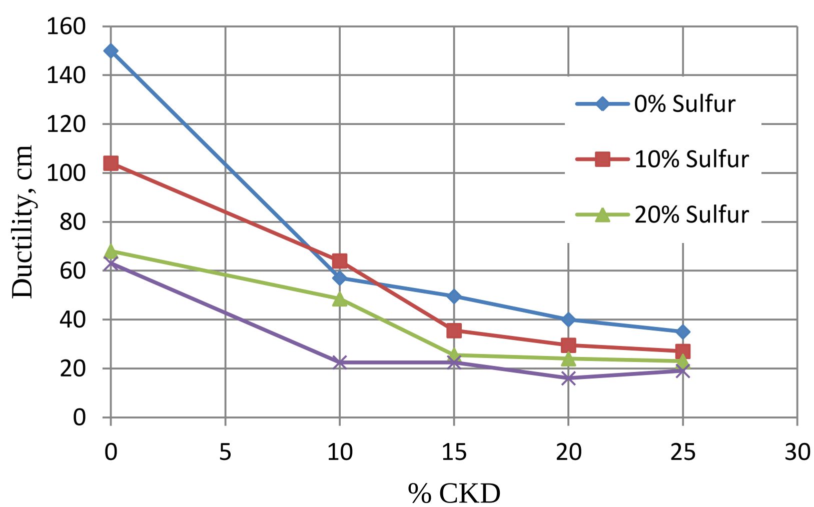

Figure 4-28: Ductility of CKD-Sulfur mastic

4.2.5 Sludge – Filler mastics

Oil sludge (OS) has a negative effect on the ductility of the asphalt material. Just like HOFA, the initial 10% of OS causes a tremendous ductility loss by about 75% when compared to the fresh asphalt, after which the rate of decrease slow down considerably, see *Figure 4-29*. Blends containing both OS and HOFA exhibits normal interaction between the two additives, the ductility decreases with more OS as well as with more HOFA uniformly.

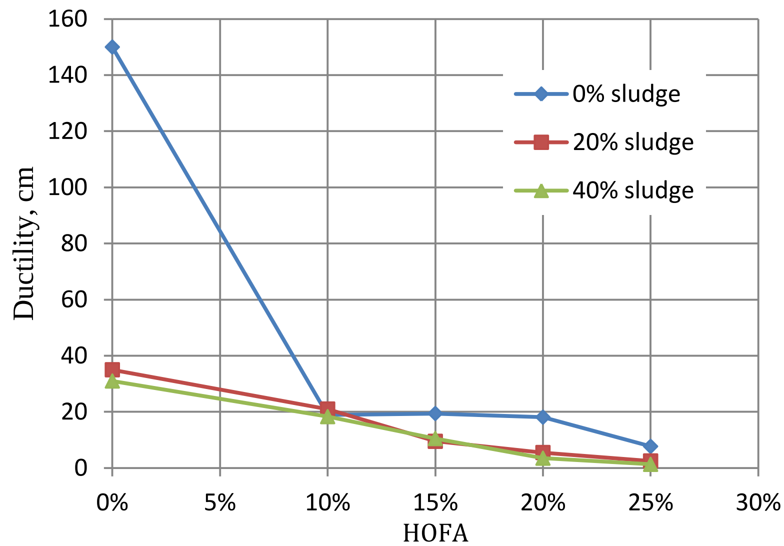

Figure 4-29: Ductility of HOFA-Sludge mastic

Normal interaction can also be observed from *Figure 4-30* for blends containing both CKD and OS additives. The ductility decreases with increase in both CKD and OS when used individually as well as in combination. This trend is maintained for the hybrid blends, with 20% and 40% curves showing a uniform transition rate in ductility.

The ductility decreases with more LMD also, for LMD-only blends, with an almost linear uniform decline trend. But when combined with OS, the LMD has little effect on the ductility in the sense that ductility value of LMD-OS blends is more or less equal to the OS-only composite. This can be seen from the 40% and 20% OS curves in *Figure 4-31*, which are approximately horizontal lines.

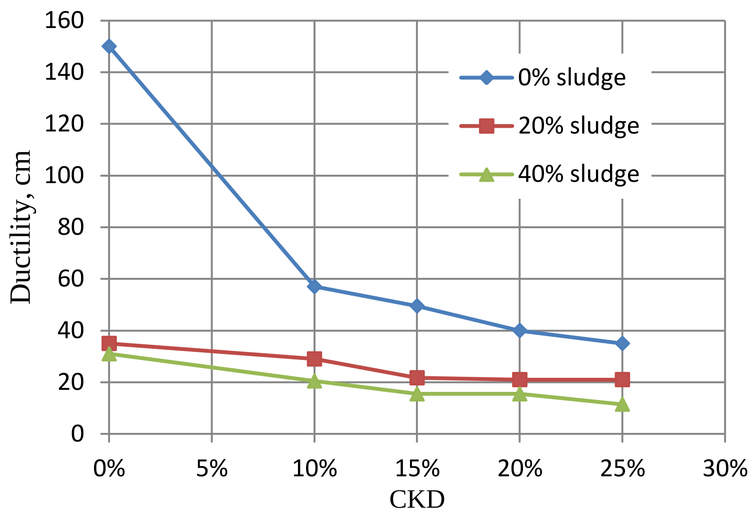

Figure 4-30: Ductility of CKD-Sludge mastic

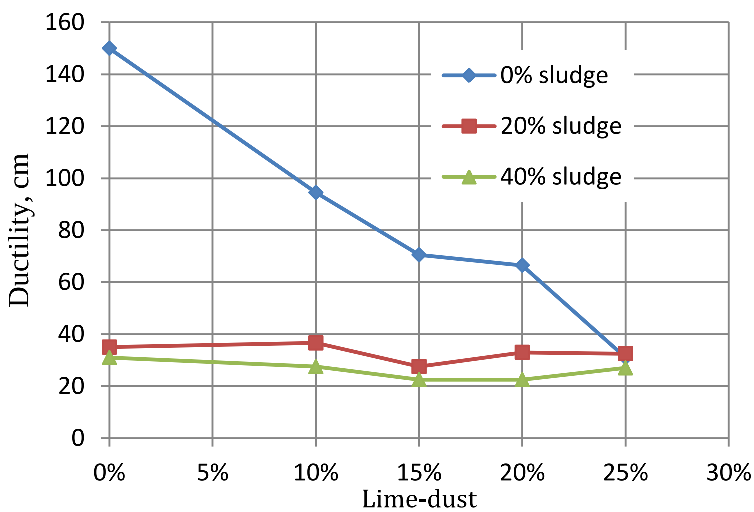

Figure 4-31: Ductility of LMD-Sludge mastic

4.3 PENETRATION

Penetration test results of the various asphalt mastics were studied in sequence according to group containing polymer, sludge, sulfur or combination of sulfur with polymer. Each of the above mastic is considered for all filler (HOFA. CKD and LMD) composition as one group. The polymer mastics were first analyzed, followed by the polymer-sulfur mastics, then the sulfur mastics result was studied before the results from sludge blends were discussed.

4.3.1 Modified Eva-Filler mastics

The penetration is seen to be increasing with the addition of modified-EVA ‘mod-EVA’, and the HOFA-mod-EVA mastics possess penetration value higher than the HOFA-only blends, as can be seen from *Figure 4-32*. However the 5% and 10% mod-EVA curves tend to demonstrate more or less similar penetration.

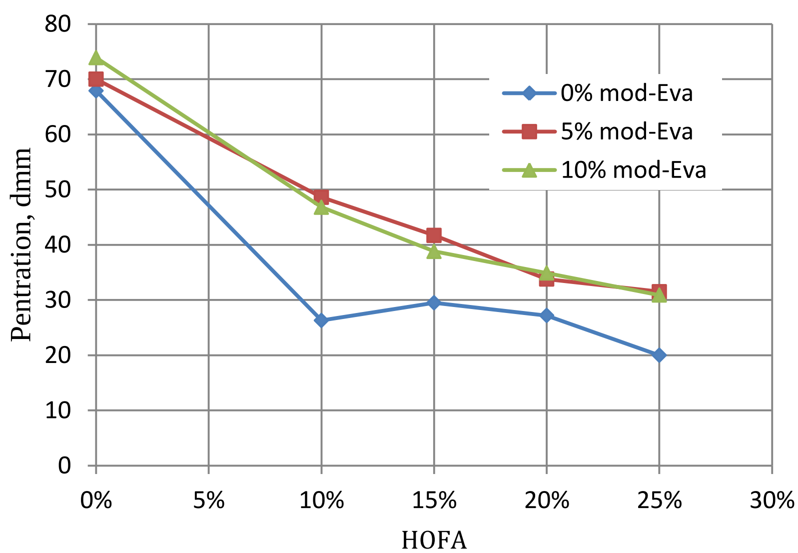

Figure 4-32: Penetration of HOFA-Mod-Eva mastic

Similar trend as in the HOFA-blends can be observes with the LMD-mod-EVA mastics from *Figure 4-33*. Only that the decline in penetration with increasing filler content is more obvious with HOFA situation.

The manner in which the mod-EVA affect the penetration differs when used along with CKD. The penetration seem to be increasing with mod-EVA for mod-EVA-only mastics, but for the double-additive blends containing both CKD and mod-EVA, the penetration increasing tendency mod-EVA is neutralized even at 10% mod-EVA content. It can also be seen from *Figure 4-34*, that the 0% and 10% mod-EVA graphs possess almost the same penetration result.

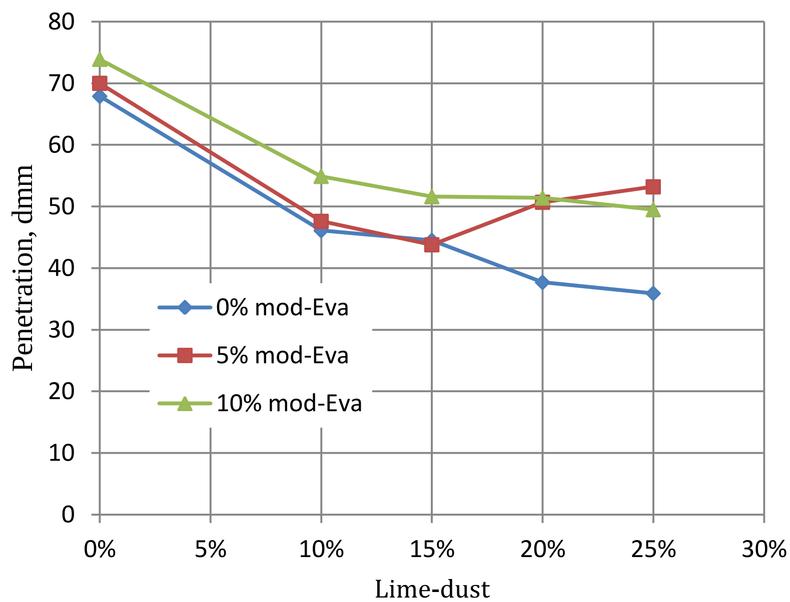

Figure 4-33: Penetration of LMD-Mod-Eva mastic

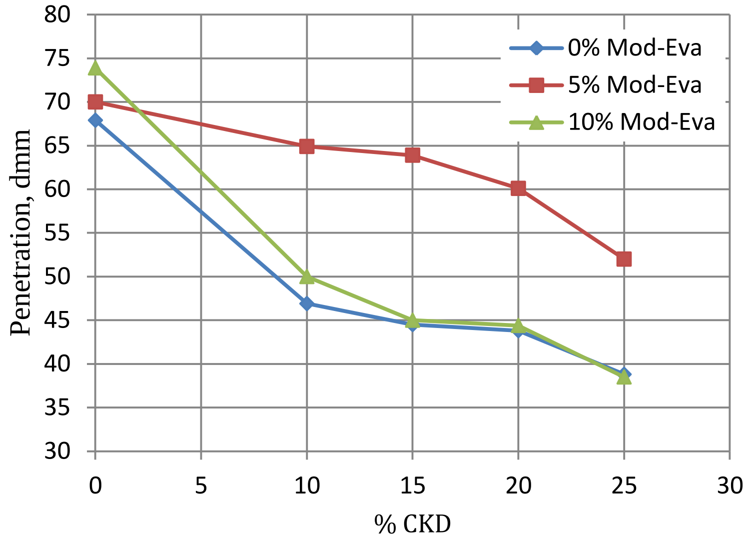

Figure 4-34: Penetration of CKD-Mod-Eva mastic

4.3.2 Styrene Butadiene Styrene-Filler mastics

The styrene-butadiene-styrene ‘SBS’ causes a large decrease in penetration value of the asphalt by more than 50% for the first 5% SBS content. As observed from previous result of the HOFA and other fillers (LMD and CKD) also, causes the penetration result to fall. But the initial addition of fillers (10-15%) to the SBS modified asphalt gives material with slightly higher penetration than the SBS only blends. As the filler content is increased (20-25%), the loss of adhesion between the polymer molecule and the asphalt matrix due to the introduction of the dry oil-absorbing filler is compensated by the closely parked arrangement of the filler grain structure within the modified asphalt resulting from their numerous quantities. This lead to the formation of composite with similar or even lesser penetration than the SBS only blends, *Figure 4-35, 4-36, & 4-37* shows some evidence regarding the above speculations.

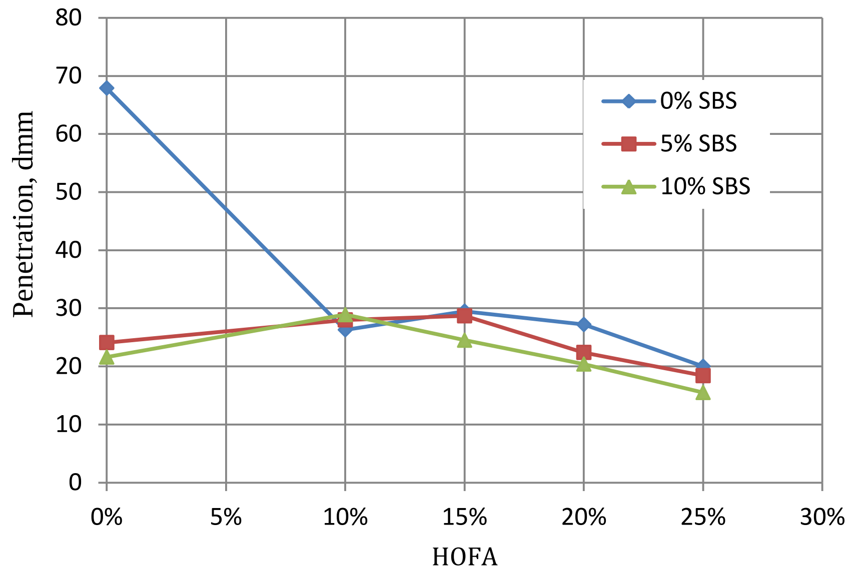

Figure 4-35: Penetration of HOFA-SBS mastic

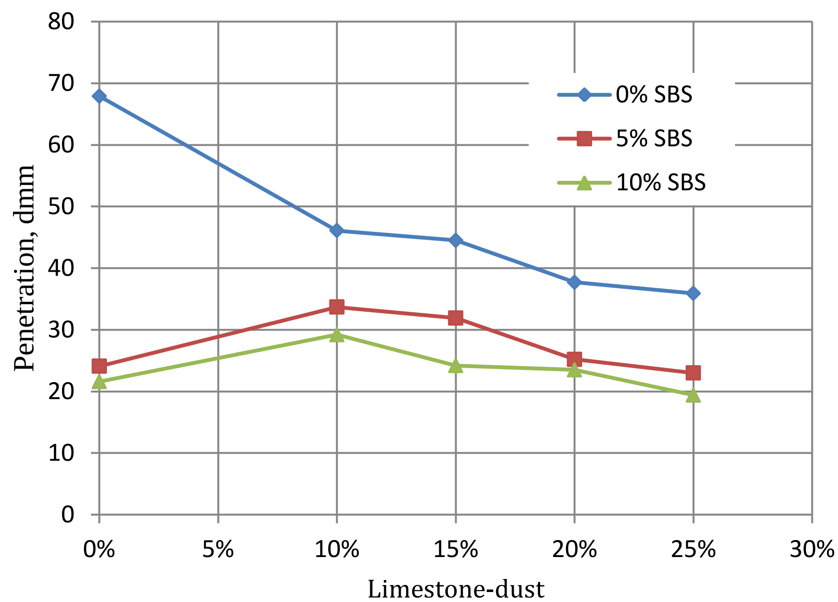

Figure 4-36: Penetration of LMD-SBS mastic

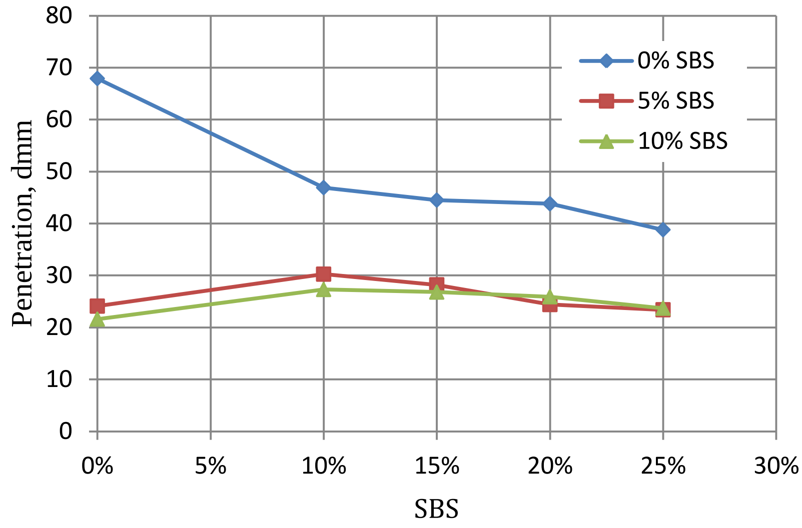

Figure 4-37: Penetration Of CKD (constant SBS graphs)

4.3.3 Mod-Eva, Sulfur and Filler-mastics

From *Figure 4-48*, the 20% and 30% sulfur blends of 5% Mod-Eva-modified asphalt is seen to have equal penetration at lower LMD composition (0 – 15%), with a slight difference for blends containing higher LMD (20 – 25%). The opposite is the case for CKD mineral filler as shown in *Figure 4-39*.

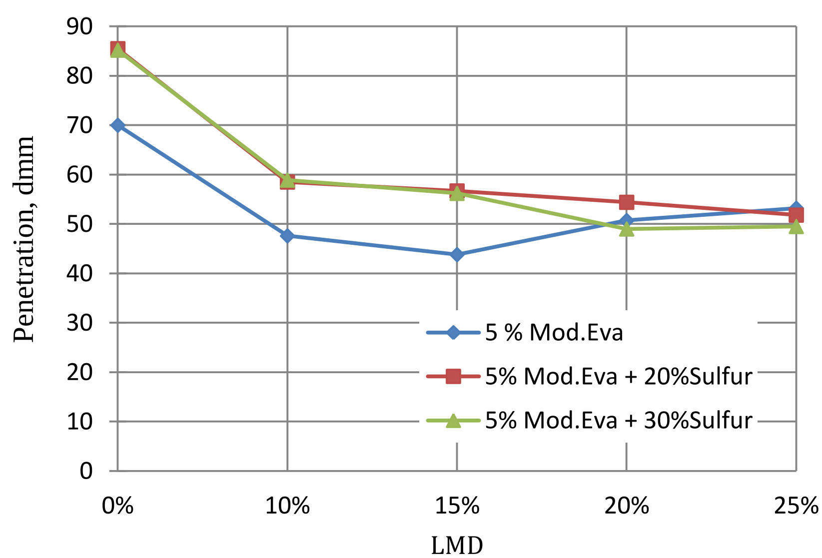

Figure 4-38: Penetration of LMD-Mod-Eva Sulfur mastic

The 30% sulfur containing 5% Mod-Eva asphalt modified blend also shows less and less penetration with higher HOFA and the 20% sulfur blends, this can seen from *Figure 4-40*. This is seen later to be as a result of the more numerous HOFA and sulfur grains.

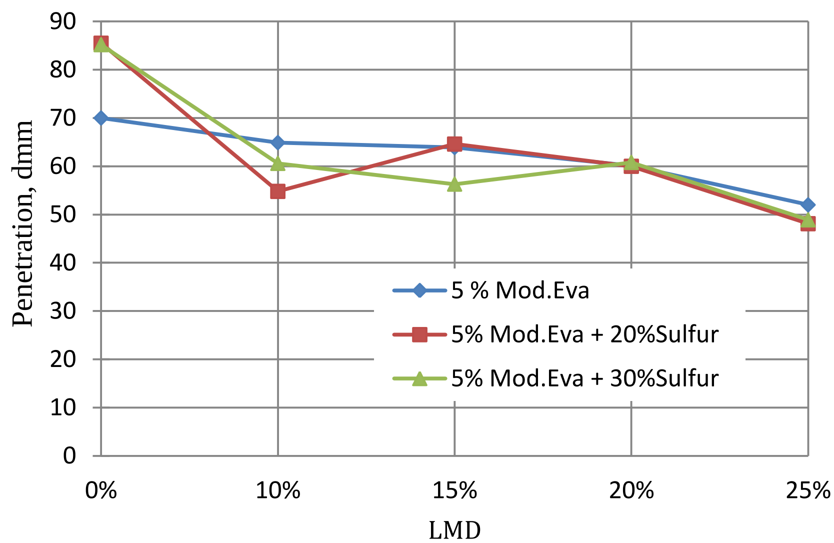

Figure 4-39: Penetration of CKD-Mod-Eva Sulfur mastic

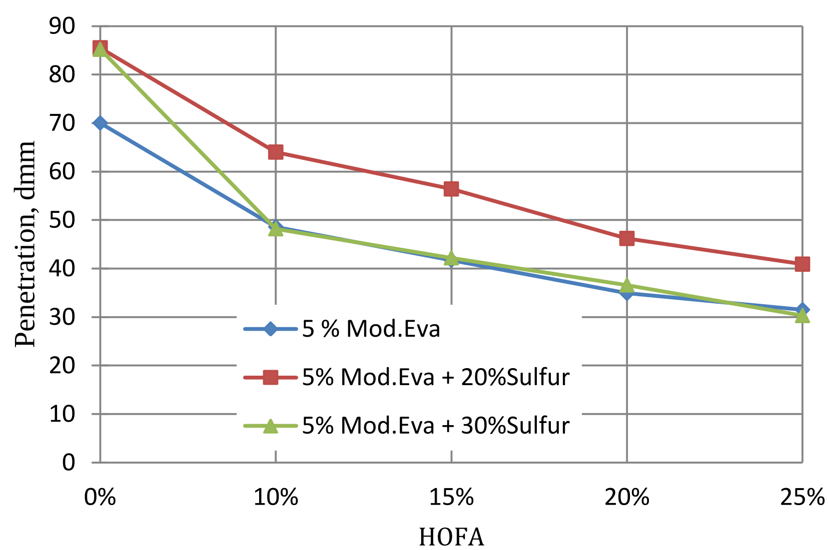

Figure 4-40: Penetration of HOFA-Mod-Eva Sulfur mastic

4.3.4 Sulfur – Filler blends

The manner in which the asphalt penetration is affected by sulfur additive depends on the amount of sulfur used to replace the asphalt component. At ranges between 0 – 10% sulfur the penetration seem to be declining with more sulfur, while beyond this interval it can be seen to be rising until it is higher than that of neat asphalt at 30% as seen from F*igure 4-41*. But a continuous decline in penetration with more HOFA is evident for HOFA-only blends.

The effectiveness of HOFA in cutting the penetration value of the asphalt has been attenuated by sulfur additive in the Sulfur-HOFA blends. Original penetration value is slightly affected for the two component blends, as shown in *Figure 4-42.*

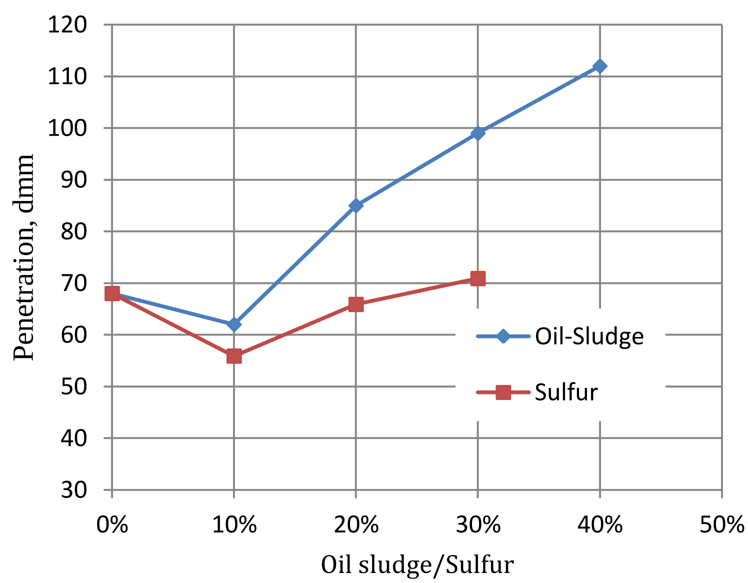

Figure 4-41: Penetration of Oil-sludge & Sulfur mastics

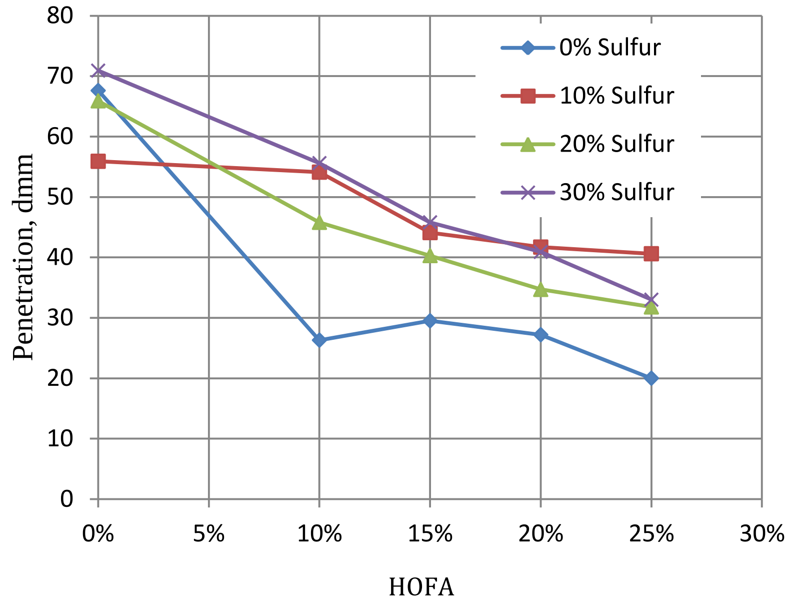

Figure 4-42: Penetration of HOFA-Sulfur mastic

Figure 4-43 shows all blends containing both sulfur and lime-dust to possess a penetration that is slightly higher or little below that of the original asphalt. The lime dust-alone containing blends show maximum penetration decrease of 30% at 10% LMD.

Similar to LMD blends, the CKD blends have an increased penetration with lower CKD, but the combination of sulfur with CKD results in higher penetration than that of pure asphalt except for 30% sulfur blend at higher content of CKD (15 – 25%) as observed from *Figure 4-44*.

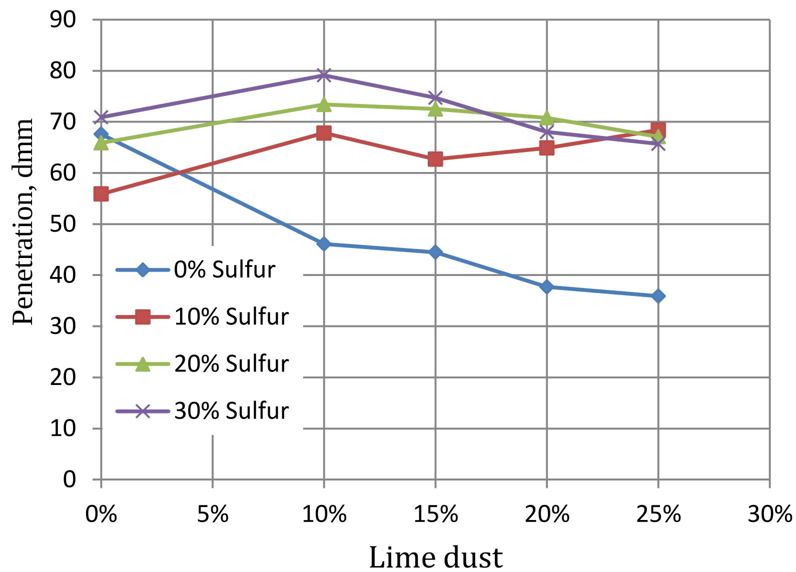

Figure 4-43: Penetration of LMD-Sulfur mastic

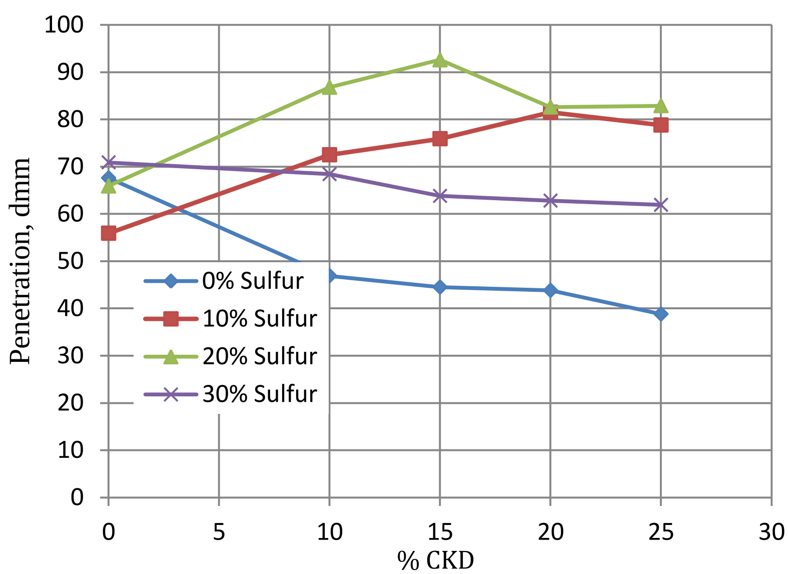

Figure 4-44: Penetration of CKD-Sulfur mastic

4.3.5 Sludge – Filler mastics

The oil sludge (OS) increase the penetration value of the asphalt composite at doses above 10%, an average uniform increase of about 20dmm for every 20% increase in OS can be observed for OS only blends from *Figure 4-41*. Below 10%, the penetration seems to be declining as in the case of sulfur. The reason behind this is attributed both the critical filler/additive content that will reverse the original asphalt matrix having the filler particles interlocked within the asphalt network (asphalt continuous phase) to asphalt patches located within the mesh of the additive (OS or Sulfur). The individual trend of both OS and HOFA were maintained even in composite containing both additives, but the decrease in penetration with increasing HOFA is more significant in blends containing high OS (40%) than those containing 20% OS, based on the graphs from *Figure 4-45*.

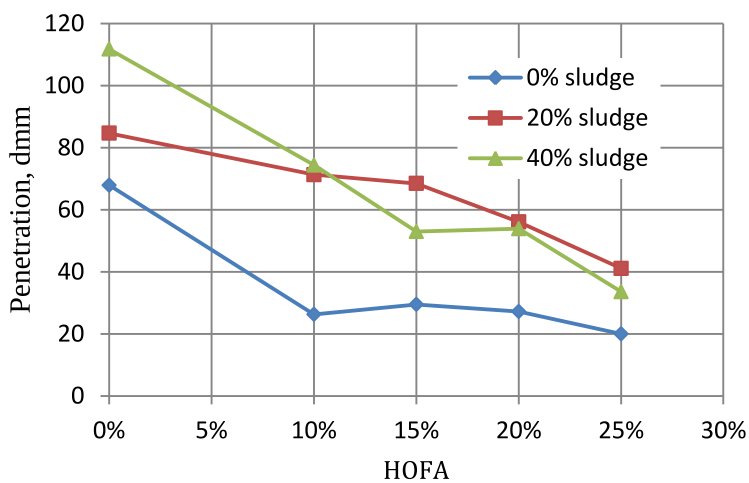

Figure 4-45: Penetration of HOFA-Sludge mastic

An abnormal rise in penetration value with increasing CKD content can be observed for 20% sludge curve in *Figure 4-46*. The same phenomenon is observed for 10% and 20%

sulfur curve for the CKD filler from *Figure 4-44*. Instead of declining with more filler content as usual, it turns out the penetration keep rising up to 20% CKD then drops a little at 25%. The possible explanation to this different trait could be the relatively higher particle size of the CKD compared to the other fillers. This will result to uneven and sparsely distributed filler-grains which produces less strong CKD-asphalt-sludge monolith having more weak asphalt-sludge three dimensional spots at lower CKD content. When the penetration needle is released, it passes through these weak spots and easily pushed downward any CKD particle blocking it path. So even when the CKD quantity increases, the result is a more weaker adhesion of the asphalt-OS fluid to the more numerous CKD grains, but as these fines are increased further, their downward displacement by the needle tend to slow, thus resulting in relatively lesser penetration value (25% CKD). But in higher sludge/sulfur content (40% sludge and 30% sulfur), the 3-dimensional matrix is more stably compact, with the sludge fine or sulfur crystals in large amount also. This results in a continuous decrease in downward and lateral displacement of the CKD grains as they are now situated in a highly filled asphalt matrix.

Similar but not obvious behavior can be observed in LMD-OS blend from *Figure 4-47*, but due to finer sizes of the LMD particles, the trend reverses immediately after 10% LMD content. The lime dust result to decline in penetration reading when added to sludge-asphalt composite, even though the effect is very slight.

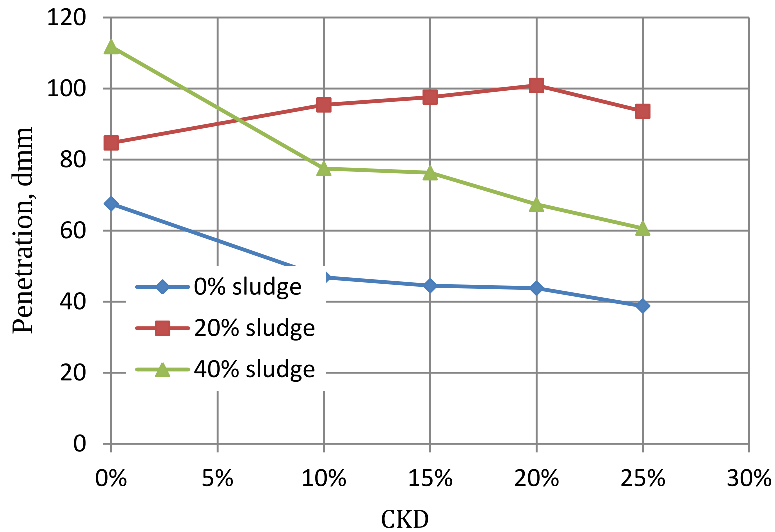

Figure 4-46: Penetration of CKD-Sludge mastic

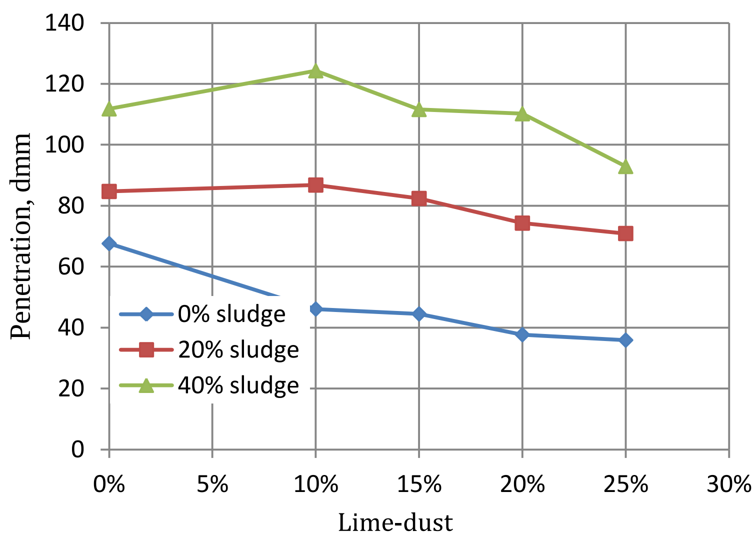

Figure 4-47: Penetration of LMD-Sludge mastic

4.4 Viscosity

Viscosity test results of the various asphalt mastics were studied in sequence according to group containing polymer, sludge, sulfur or combination of sulfur with polymer. Each of the above mastic is considered for all filler (HOFA, CKD and LMD) composition as one group. The polymer mastics were first analyzed, followed by the polymer-sulfur mastics, then the sludge mastic results was studied before the results from sulfur blends were discussed.

4.4.1 Modified-EVA-Filler mastics

The viscosity increases with more modified-EVA 'mod-EVA' as well as with the HOFA, as seen from *Figure 48.*

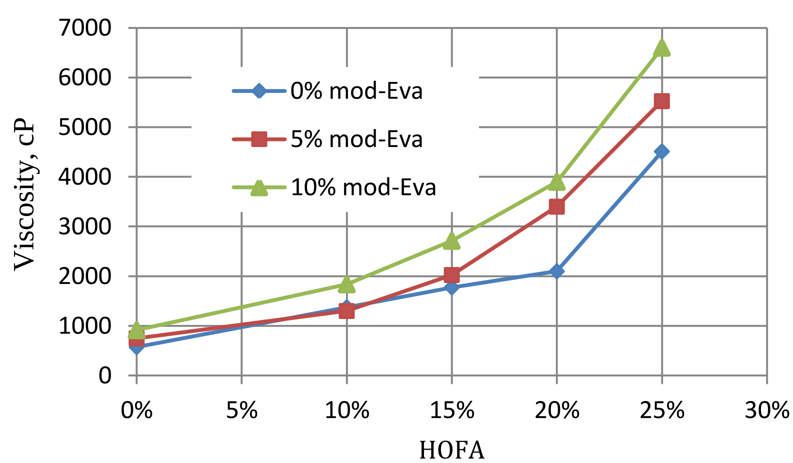

Figure 4-48: Viscosity of HOFA-Mod-Eva mastic

Moderate increase in viscosity can also be observed for the modified-EVA-LMD mastics with both additives increase from *Figure 4-49*, as with HOFA-mod-EVA blends (*Figure 48)*. The only difference here is that the viscosity increase linearly with higher LMD filler content as opposed to the exponential trend depicted by HOFA filler.

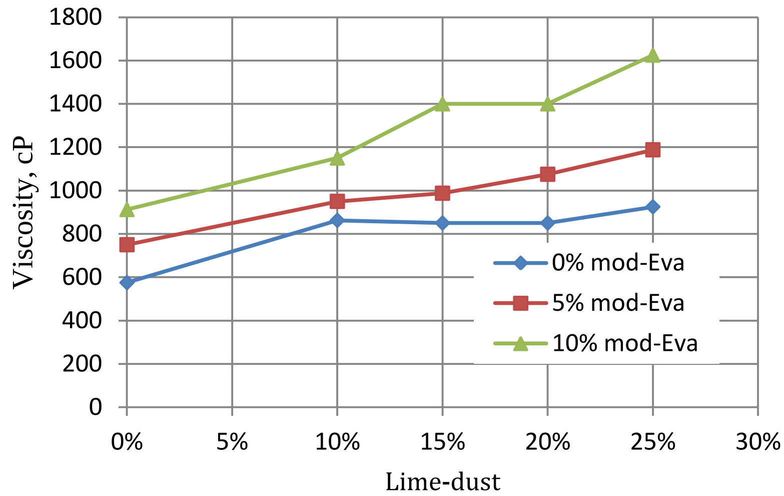

Figure 4-49: Viscosity of LMD-Mod-Eva mastic

The mod-EVA mastics tend to thicken more when blended with CKD filler as compared to LMD, as shown in *Figure 4-50*, the viscosity value for the same filler content can be seen to be higher for CKD.

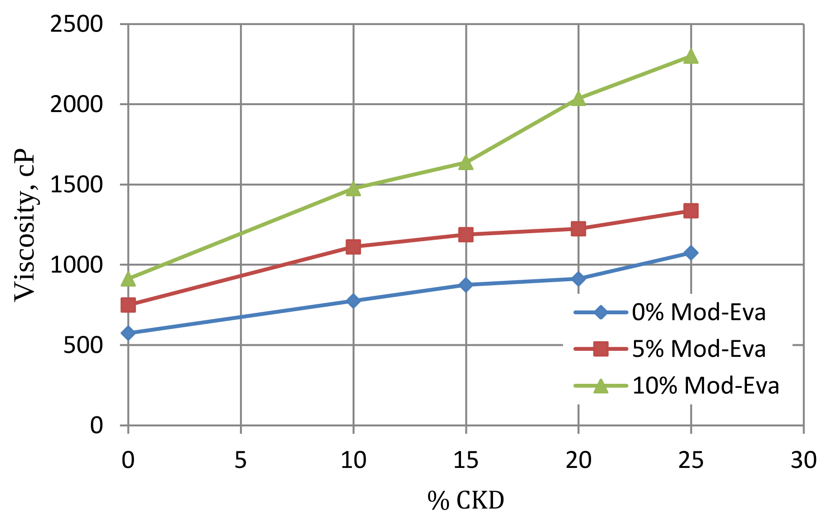

Figure 4-50: Viscosity of CKD-Mod-Eva mastic

4.4.2 Styrene Butadiene Styrene-Filler mastics

The SBS polymer tend to yield a highly viscous material that when further mixed with filler result in material with viscosity beyond the measurable range of the experimental set-up (20 rpm, 135°C) at any percent composition of the HOFA for the 5% and 10% SBS content. But for the LMD and CKD, similar viscous materials are produce, only in this case within the measurable range of the test set-up for the 5% SBS modified at all filler content (10-25%). The 10% SBS modified asphalt produce composite of high immeasurable viscosity for the entire filler content range.

4.4.3 Mod-EVA, Sulfur and Filler mastics

The sulfur containing 5% Mod-EVA modified asphalt mastic exhibit lower viscosity than the 5% Mod-EVA – only blend almost throughout the whole range of fillers (HOFA, CKD, LMD) content, but with the 30% sulfur showing a slightly higher viscosity relative to the 20% sulfur blend. The earlier expected trend has been retained, see *Figure 4-51, 4-52, 4-53* for more detail.

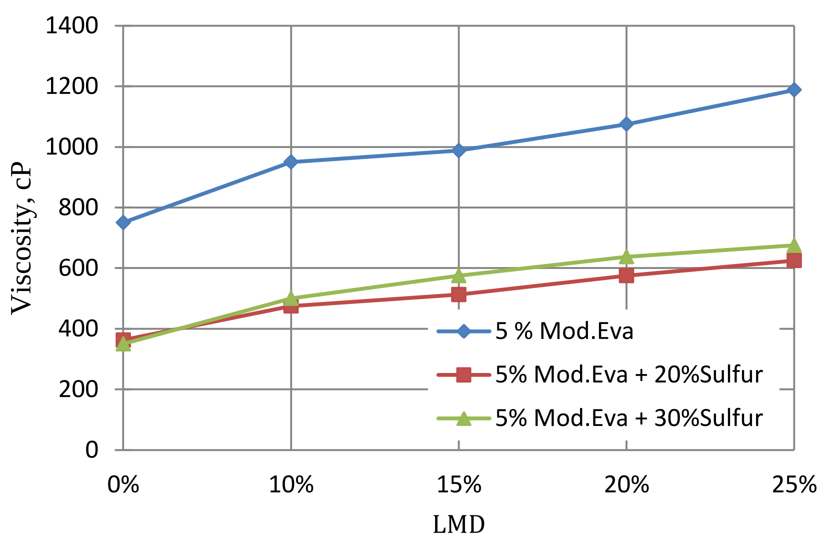

Figure 4-51: Viscosity of LMD-Mod-Eva Sulfur mastic

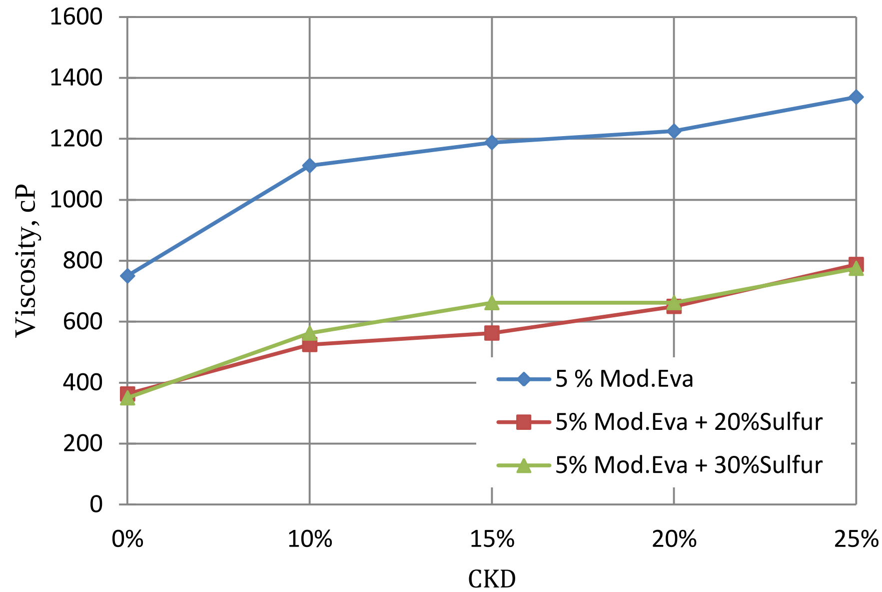

Figure 4-52: Viscosity of CKD-Mod-Eva Sulfur mastic

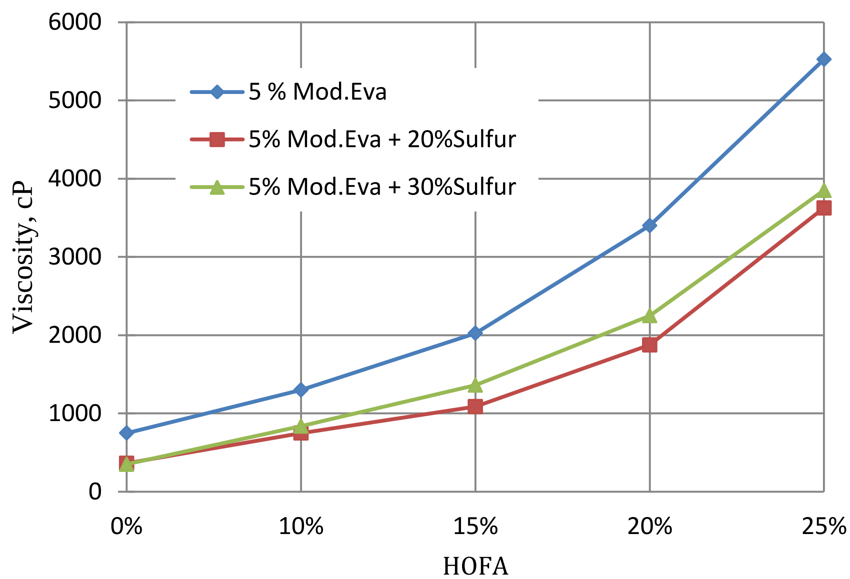

Figure 4-53: Viscosity of HOFA-Mod-Eva Sulfur mastic

4.4.4 Sulfur – Filler mastics

The addition of sulfur to neat asphalt causes a gradual drop in its viscosity. Initially (at 10%) the effect is minimal with just a decrease of about 2%, then followed by a significant drop of more than 40% at 20% sulfur, as shown in *Figure 4-57*. on the other hand HOFA shows a tremendous thickening ability, which could be attributed to its ability to absorb the oily constituent of the asphalt that in turn result to higher interlayer friction. 10% HOFA leads to about 200% rise in viscosity. Mixing sulfur with the HOFA-blend brought the viscosity close to the original value especially within 20-30% sulfur and 10-15% HOFA ranges combination, refer to *Figure 54.*

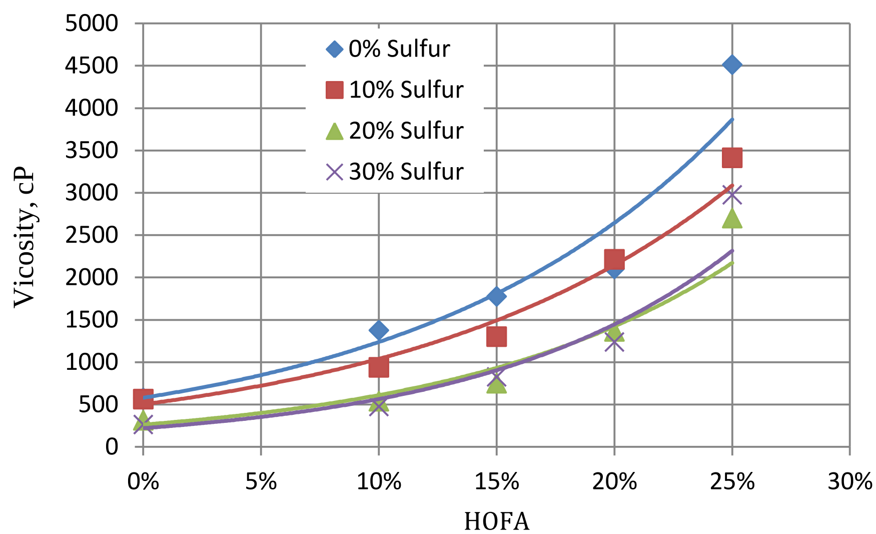

Figure 4-54: Viscosity of HOFA-Sulfur mastic

The limestone dust does not seem to change the viscosity significantly but an increase of 300cP could be noticed for the first 10% LMD from *Figure 4-55*. Afterward there seem to be no change up to 20% LMD content. Adding sulfur causes the viscosity to go down below the normal asphalt viscosity, this is observed for all sulfur containing LMD-asphalt mastic.

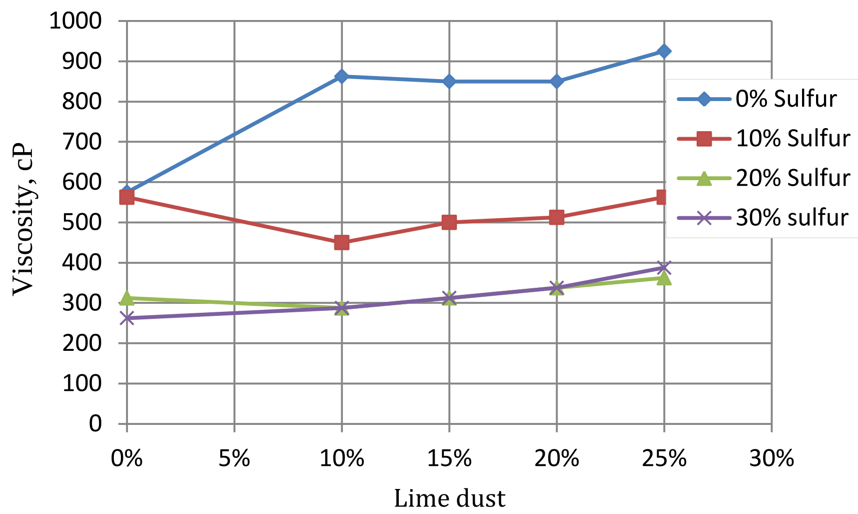

Figure 4-55: Viscosity of LMD-Sulfur mastic

And the CKD blends more or less behave in similar manner as the LMD mastics, the sulfur containing CKD samples possesses lower viscosity than the neat bitumen as seen from *Figure 4-56*, for CKD-only mastics the viscosity appreciates but not considerably as in HOFA.

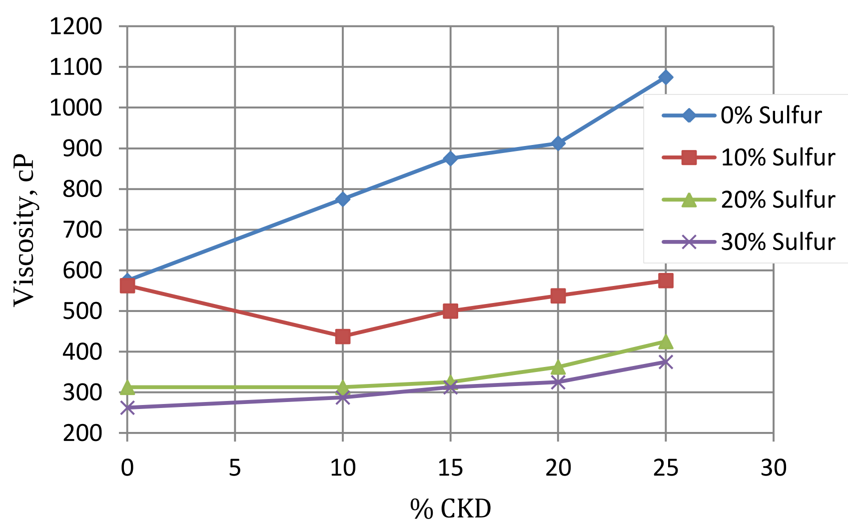

Figure 4-56: Viscosity of CKD-Sulfur mastic

4.4.5 Sludge – Filler mastics

The Oil-sludge (OS) has little effect on the asphalt viscosity as previously observed with most of the asphalt properties. The behavior pattern differs below and above 10% sludge content for sludge-only blends, see *Figure 4-57*. The viscosity tend to be increasing with more sludge for doses below 10%, after which it starts declining until it falls a bit below the original asphalt viscosity at 40% OS content by 37.5 centi-poise. This also is attributed to the phase inversion that occurs at OS content above 10% but below 20% OS.

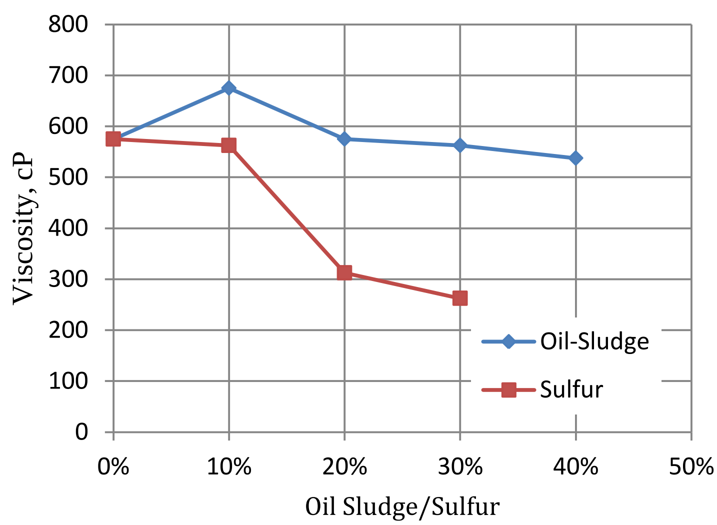

Figure 4-57: Viscosity of Oil-sludge & sulfur mastics

Figure 4-58 shows the viscosity of HOFA-OS blends, the resulting viscosities are always higher than that of HOFA-only mastics, unlike in the case with sulfur. This signifies a constructive interaction between the two additives. The increase trend is exponential as it is with the HOFA-only blends.

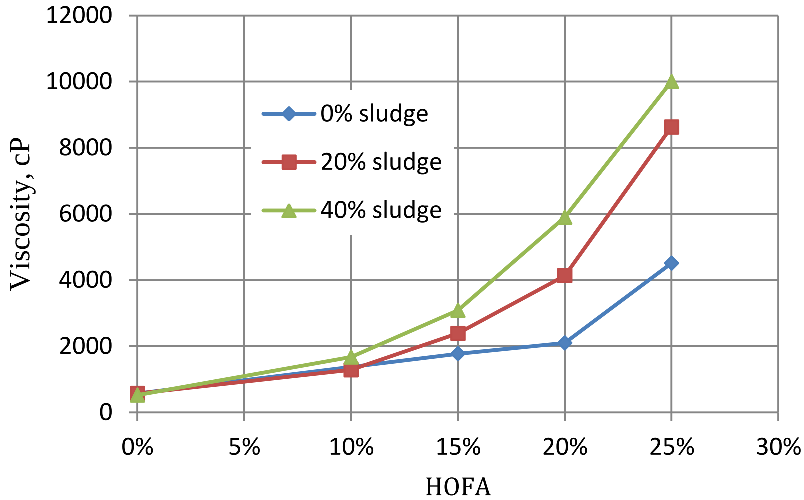

Figure 4-58: Viscosity of HOFA-Sludge mastic

The viscosity also continues to rise with more CKD and OS for CKD-OS blends, a uniform trend can be observed from *Figure 4-59*. On the contrary, the viscosity value tends to be decreasing with higher OS content for LMD–OS blends as opposed to CKD-OS mastics. But *Figure 4-60 shows* the linear increasing trend with more LMD to be maintained.

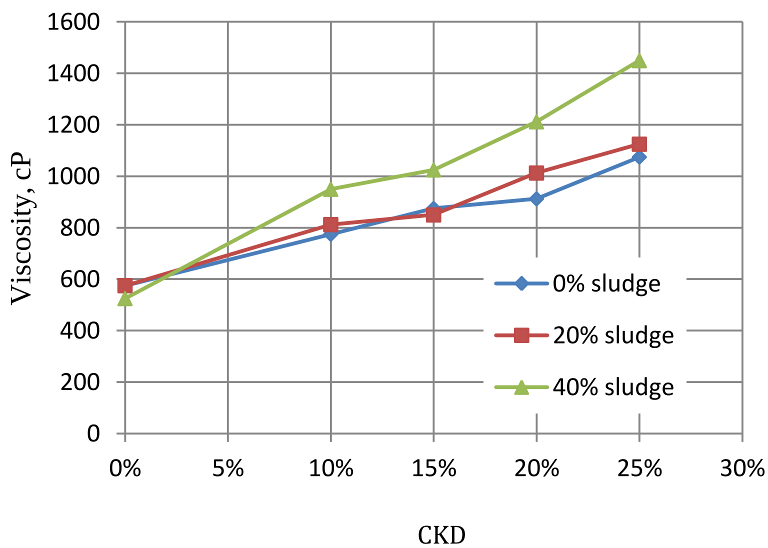

Figure 4-59: Viscosity of CKD-Sludge mastic

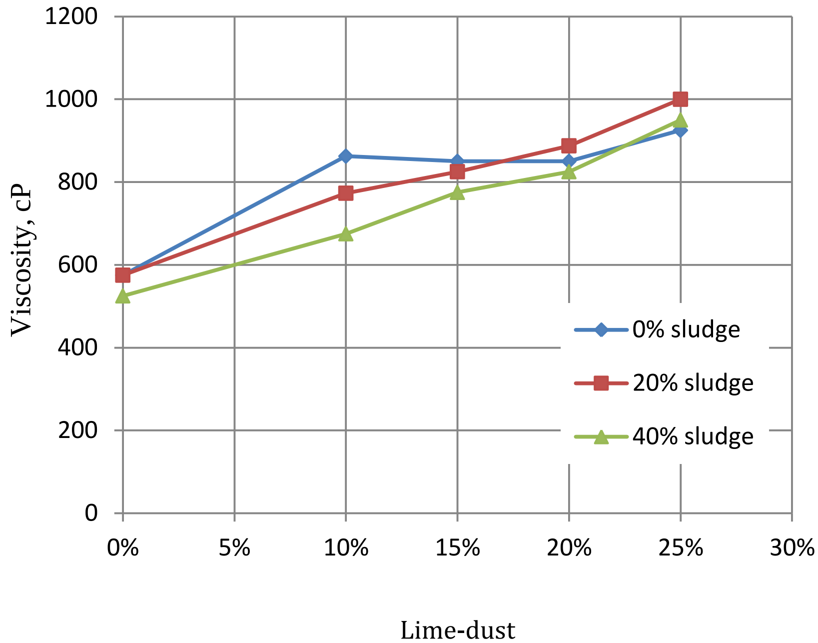

Figure 4-60: Viscosity of LMD-Sludge mastic

4.5 Flash Point (FP)

Flash point test results of the various asphalt mastics were studied in sequence according to group containing polymer, sludge, sulfur or combination of sulfur with polymer. Each of the above mastic is considered for all filler (HOFA. CKD and LMD) composition as one group. The sulfur mastics were first analyzed, followed by the sludge mastics, then the polymer blends result was studied before the results from polymer-sulfur blends were discussed.

4.5.1 Sulfur – Filler mastics

The mineral fillers and HOFA has little or no effect on the flash point, the maximum difference between neat asphalt value and the highest filler content (25%) is not more than 15^{o}C which is very little compared to that 340^{o}C. But on the contrary due to high flammable nature of sulfur, it results in more than 100^{o}C decrease for just 10% composition. But beyond this value the rate of decline ceases to about 5^{o}C for every 10% more as can be seen from *Figure 4-61*, which is virtually insignificant.

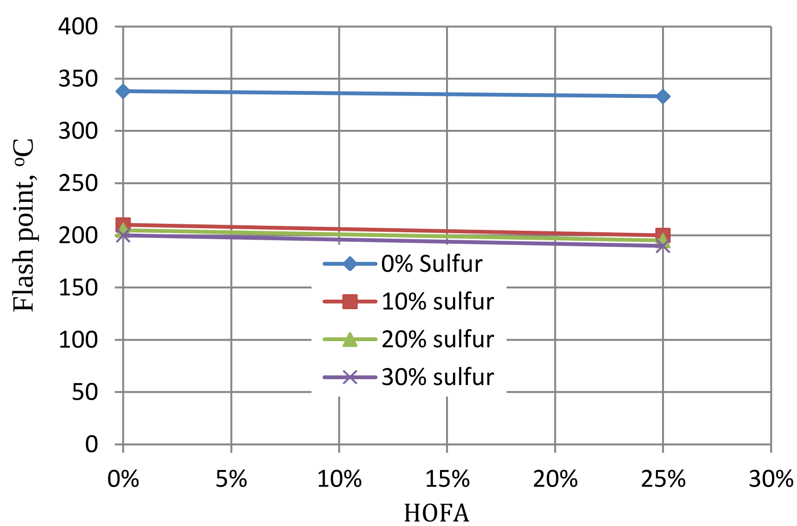

Figure 4-61: Flash point of HOFA-Sulfur mastic

Similar trend as in the HOFA-Sulfur blend is observed from *Figure 4-62*, for the LMD-Sulfur mastics. But the horizontal change in flash due to filler increase tend to be a little more pronounced with the CKD as compared to the other two additives (HOFA and LMD) especially for the CKD-only blends, as depicted by *Figure 4-63*.

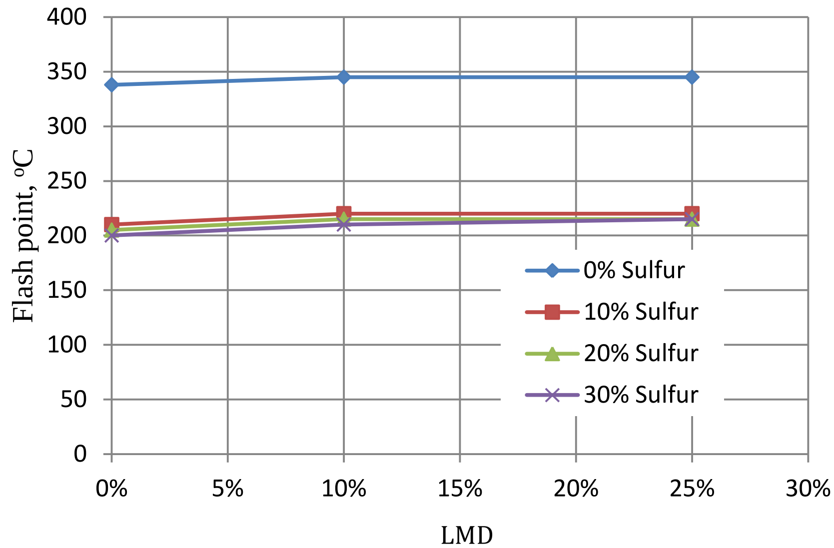

Figure 4-62: Flash point of LMD-Sulfur mastic

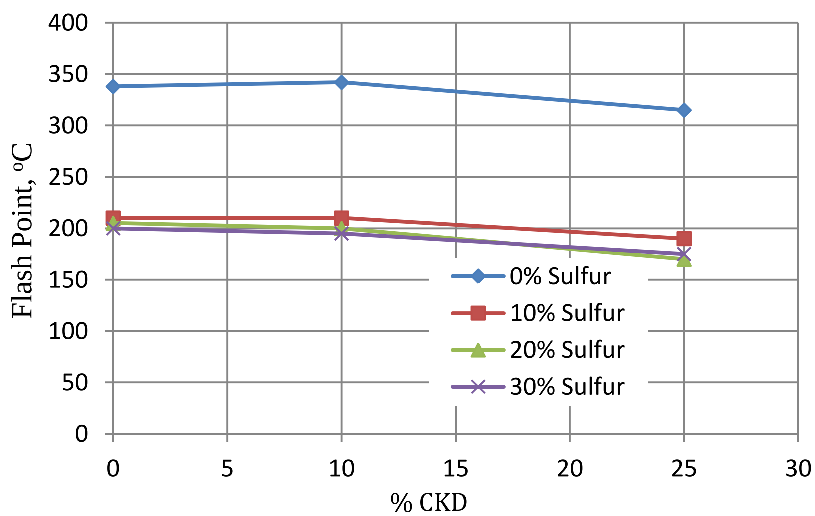

Figure 4-63: Flash point of CKD-Sulfur mastic

4.5.2 Sludge – Filler mastics

The oil-sludge (OS) has decreasing effect on the flash point (FP) of the asphalt, 20% content result to a decline of about 80^{o}C, but this effect becomes less pronounce with more additional OS, as observed from *Figure 4-64*. As for the OS-HOFA composites, the flash point almost remained unaffected with HOFA addition when compared to the OS-only mastic. This has also been observed for sulfur-HOFA mastics.

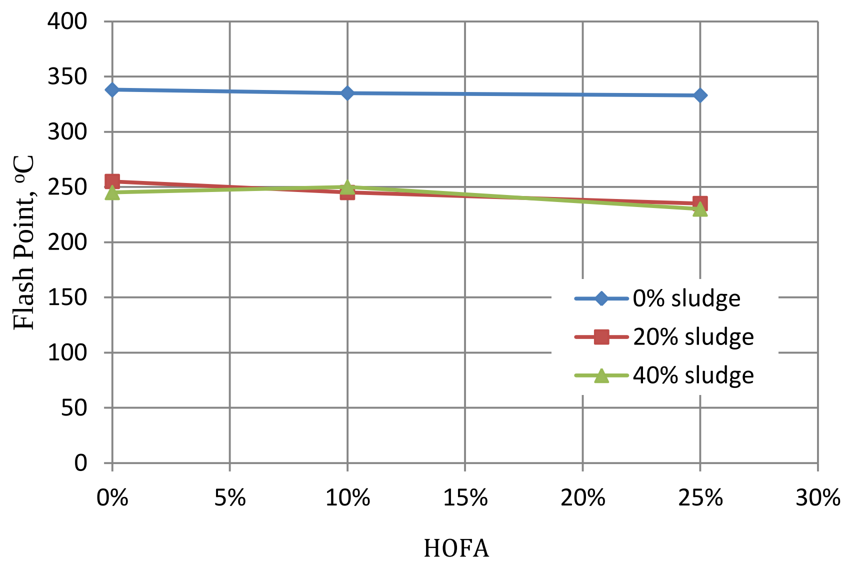

Figure 4-64: Flash point of HOFA-Sludge mastic

The CKD tends to affect the flash point more significantly in OS-asphalt medium if compared to HOFA, an average decrease of about 30^{o}C can be observed for 20% and 40% OS curve at 10% and 25% CKD contents, refer to *Figure 4-65*. All in all, there seem to be little interaction between the substances.

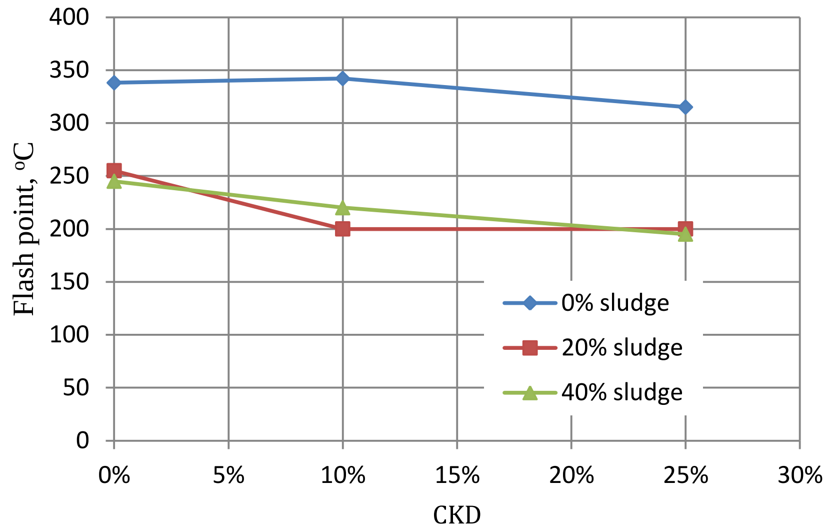

Figure 4-65: Flash point of CKD-Sludge mastic

Adding LMD to OS-asphalt blend does not seem to change its original flash point value by more than 15^{o}C for 25% content, as seen from *Figure 4-66*. The 20% and 40% OS curves are almost straight lines if one observes.

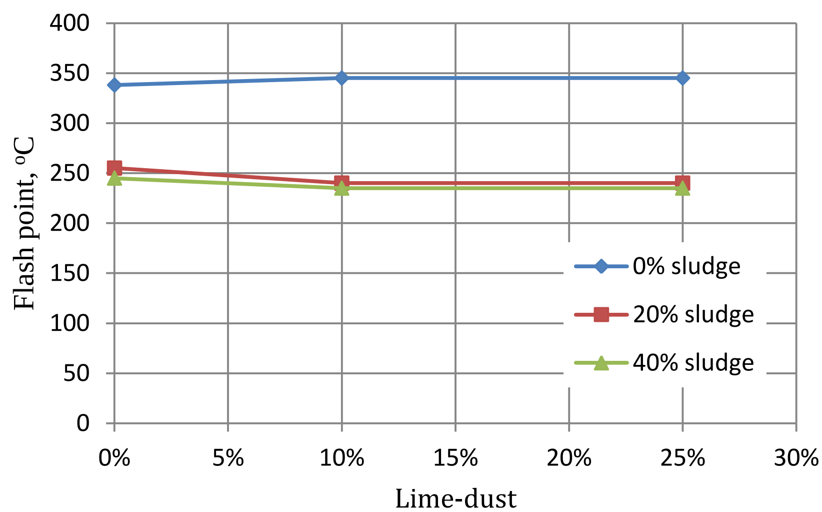

Figure 4-66: Flash point of LMD-Sludge mastic

4.5.3 Modified-EVA-Filler mastics

As previously witnessed with sulfur and sludge asphalt, the modified-EVA ‘mod-EVA’ has tremendous negative effect on the flash point ‘FP’ of the asphalt material, which can be largely attributed to its sulfur constituent. However, this significant change is restricted to the initial 5% mod-EVA content. While beyond this range the FP seem to be insensitive to any further mod-EVA addition as observed from *Figure 4-67.*

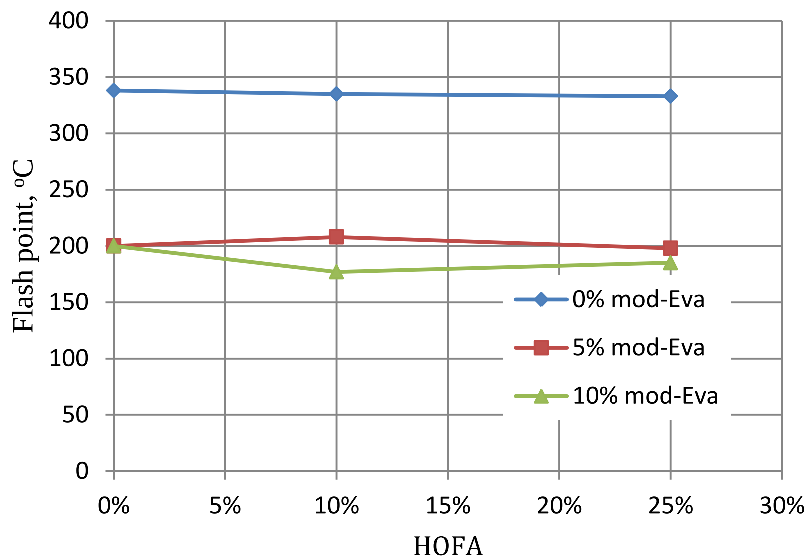

Figure 4-67: Flash point of HOFA-Mod-Eva mastics

Also like in the sulfur and sludge asphalt, the fillers have no significant effect on the FP, it can be seen from the graphs below in *Figure 4-68, and 4-69*.

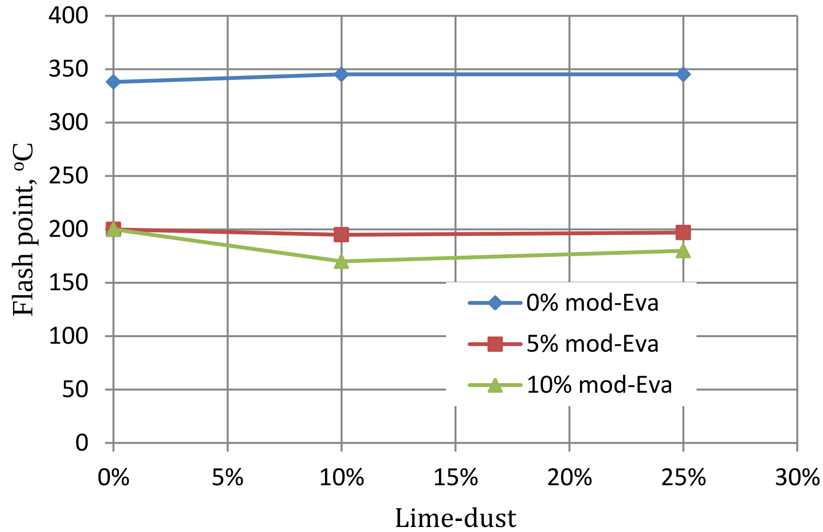

Figure 4-68: Flash point of LMD-Mod-Eva mastics

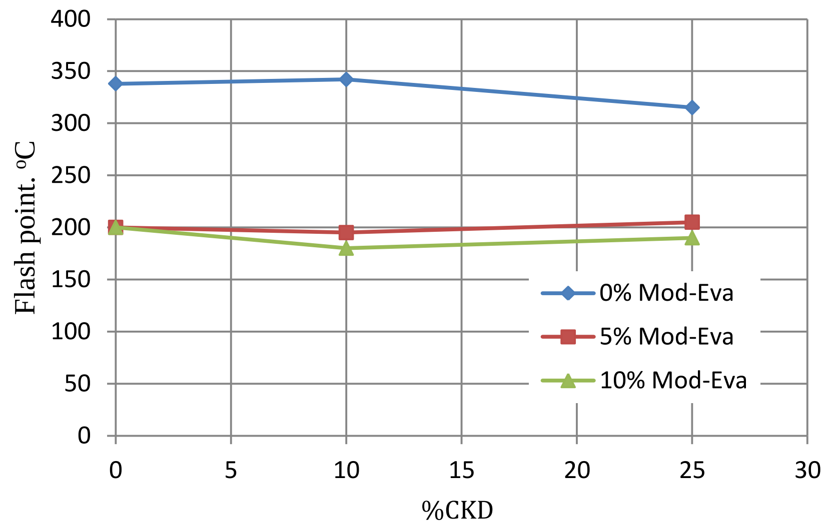

Figure 4-69: Flash point of CKD-Mod-Eva mastic

4.5.4 Styrene Butadiene Styrene-Filler mastics

The SBS polymer has little or no effect on the flash point 'FP' of the asphalt material, both the 5% and 10% SBS modified asphalt shows FP value of 330^{o}C, which is more or less equal to that of the neat asphalt (340^{o}C). It was also observed previously that the filler has little on the FP value for sulfur and sludge asphalt mastics. There seem to be relatively more pronounce fluctuation of the FP as the filler content increase if compared to the past observations, nevertheless the effect is still minor. The mentioned detail can be confirmed from *Figure 4-70, 4-71, & 4-72*.

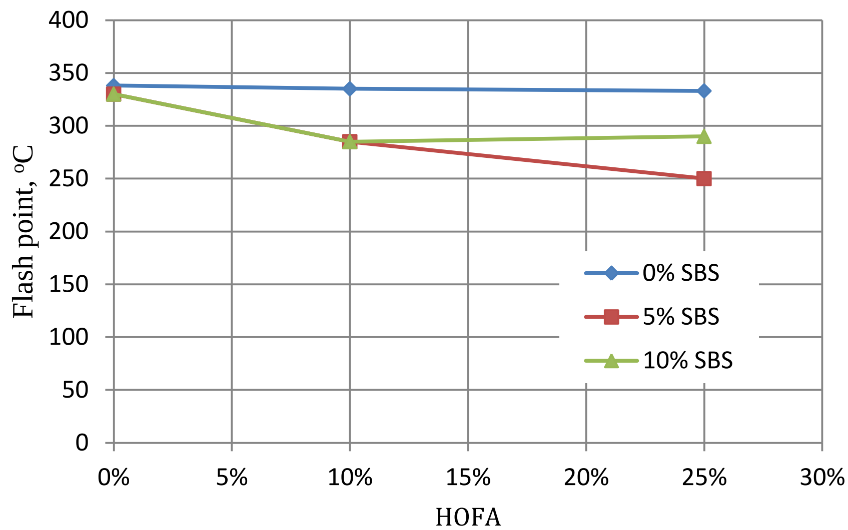

Figure 4-70: Flash point of HOFA-SBS mastic

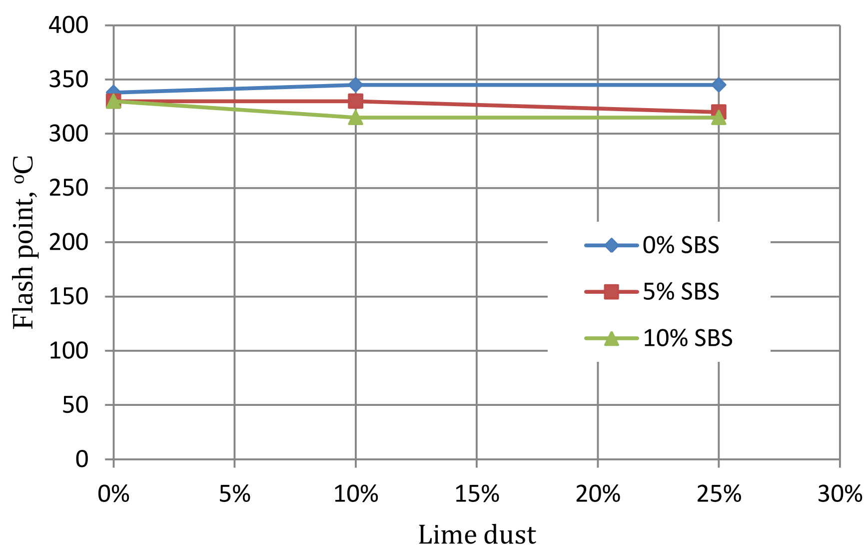

Figure 4-71: Flash point of LMD-SBS mastic

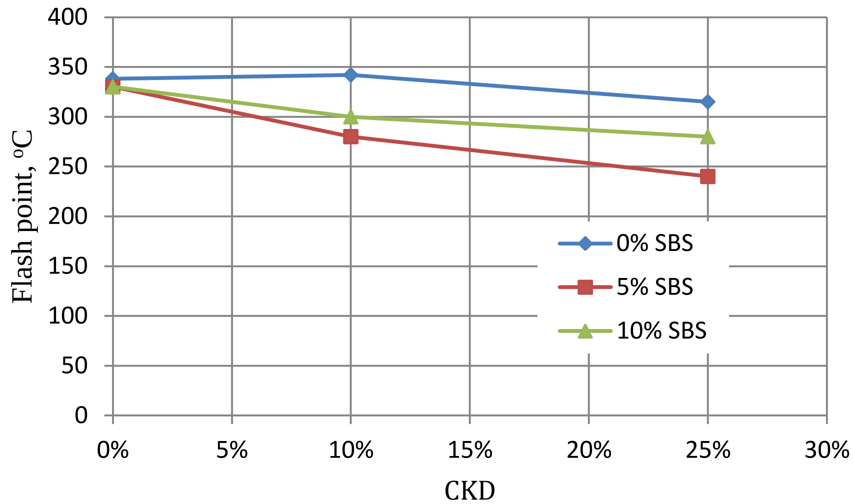

Figure 4-72: Flash point of CKD-SBS mastic

4.5.5 Mod-EVA, Sulfur and Filler mastics

Adding sulfur to the 5% Mod-EVA blend tends to further decrease its flash point value. And in contrast with the 5% Mod-EVA blend, the fillers show a relatively much more positive influence on FP, an average increase of 25°C, 20°C and 40°C at 25% content can be observe for LMD, CKD and HOFA fillers respectively from *Figure 4-73, to 4-75*.

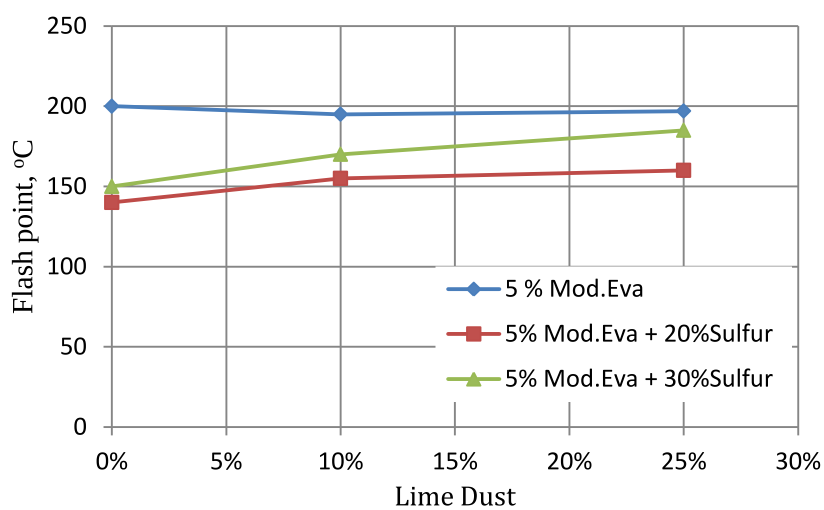

Figure 4-73: Flash point of LMD-Mod-Eva Sulfur mastic

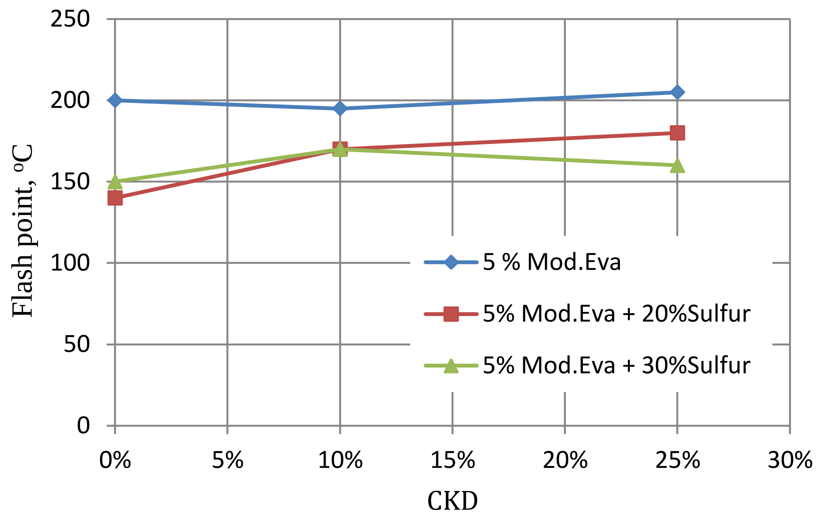

Figure 4-74: Flash point of CKD-Mod-Eva Sulfur mastic

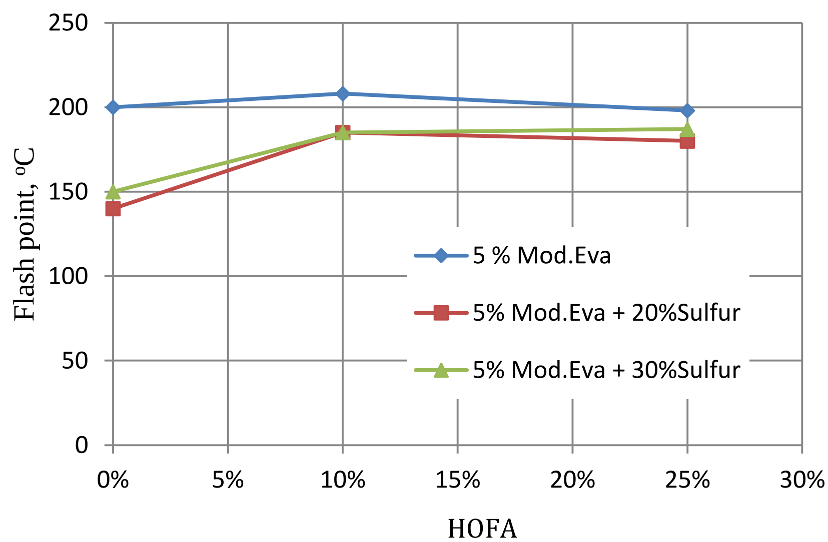

Figure 4-75: Flash point of HOFA-Mod-Eva Sulfur mastic

4.6.0 SULFUR – SBS mastics

Initially, 5% SBS with 20% and 30% sulfur blends for all the various fillers and their composition range (0 – 25%) were designed. But after the first 20% sulfur + 5% SBS polymer mastic was prepared, it became obvious that the previous design cannot be practically assessed given the type of test to be carried out, due to a process known as vulcanization. The sulfur atom tend to chemically react with the SBS polymer, forming a cross-link between the polymer chain which lead to the evolution of a highly elastic sticky non-flowing asphalt composite that is very difficult and nearly impossible to handle when it comes to sample preparation and casting in the molds, even at the initial stage of the reaction. The vulcanization temperature is a little above 150^{o}C, and mixing the SBS-asphalt or SBS-filler-asphalt blend at temperature below 150^{o}C is another issue which leads to the formation of non-homogeneous composite with unspecified characteristics due to the high softening point of SBS-asphalt mastic. This makes the option of exploring the vulcanizing advantage of sulfur-SBS more appealing, so 5% SBS with 10% and 5% sulfur blends and 3% SBS with 5% sulfur mastics were prepared and tested.

The 5% SBS + 5% sulfur materials has about 90% elongation recovery, accompanied by softening points and viscosities a little less or equal their 5% SBS-only counterparts mastics, with also penetration and ductility slightly higher than the 5% SBS-only blends. ‘3% SBS + 5% sulfur’ samples show an average recovery of 40% – 50%, their softening points values are averagely 20^{o}C less than the ‘5% SBS + 5% sulfur’ mastic, they also possess moderate viscosity. The penetration and ductility reading are also little lesser than those of the 5% SBS – 5% sulfur blends'.

4.7.0 BOND STRENGTH (BS)

Both sulfur and oil sludge affects the asphalt bond strength (BS) in similar manner, as can be seen from *Figure 4-76*. BS increase with either sulfur or Oil sludge initially, then, it starts to decline at higher content. At small percent composition, Oil-sludge particles serve as filler reinforcement engulf in sufficiently thick asphalt network surrounding them while most of the sulfur additives got attached to naphthenic constituent of the asphalt forming extra asphaltene in the process [38], which is responsible for asphalt hardening. But as the these sludge particle increase in number, the asphalt network responsible for binding them thin-out, which result to phase inversion gradually. And the amount of unreacted sulfur increases in the asphalt medium. This leads to the continuous deterioration of the composite BS.

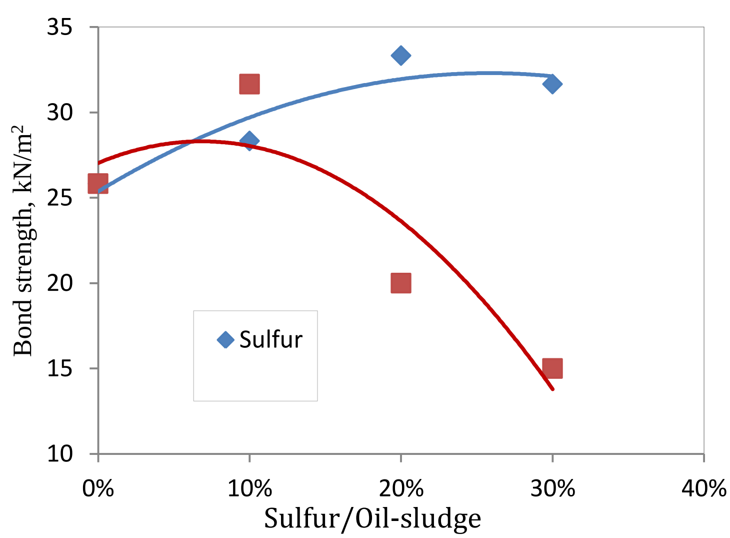

Figure 4-76: Bond strength of Sulfur & Oil-sludge mastics

The SBS causes a linear increment in the asphalt's bond strength while Mod-EVA shows slight or no improvement in BS, as shown in *Figure 4-77*.

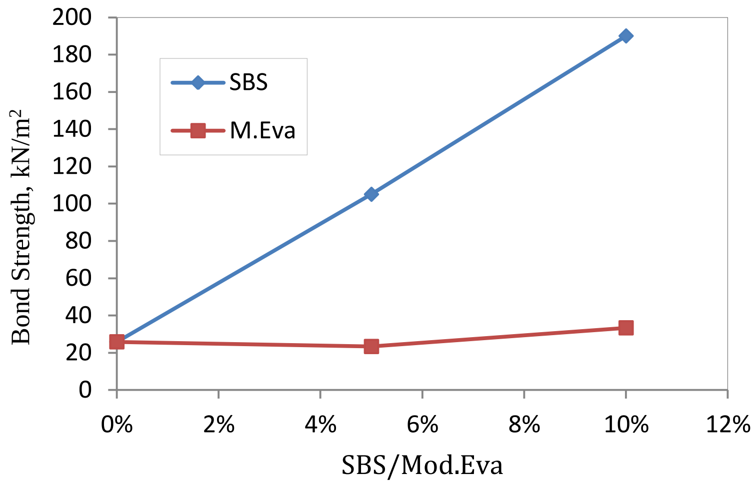

Figure 4-77: Bond strength of SBS & Mod-Eva mastics

The neat asphalts bond strength (BS) is slightly above 25kN/m^2, adding filler to the asphalt generally results to an increase in BS, as shown in *Figure 4-78*. Both CKD and LMD have produced composites with atleast 100% increase in bond strength compared to the original asphalt at 25% content, 25% HOFA yield material with 12 times BS of the pure asphalt.

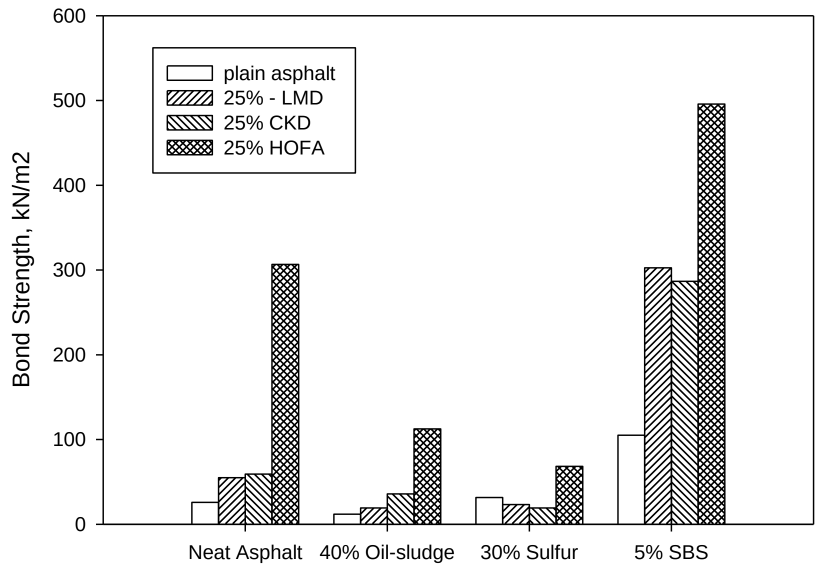

Figure 4-78: Bond strength of SBS, Sulfur & Oil-sludge mastics

The SBS-modified asphalt exhibit almost twice the bond strength possessed by the 25% CKD and LMD containing asphalt at 5% SBS content. Adding CKD or LMD to the 5% SBS blend nearly triples its bond strength, but the HOFA yet results to more than just triple as can be seen from *Figure 4-78* above**.** The 30% sulfur blend has little additional BS, but adding 25% of CKD and LMD result in material with lesser BS than the neat asphalt, even the HOFA have little effectiveness in raising the BS value at 30% sulfur content, with an increase of no more than 43 kN/m^2. 40% sludge yield composite with a very little BS (12 kN/m^2), and adding 25% filler seem not to help improve the BS much, with only the HOFA mastic having BS a little above 100 kN/m^2.

The addition of sulfur to the SBS-modified asphalt results in material with high elastic and recovery property. It can be observed form *Figure 4-79* that, *at* 5% SBS and 5%

sulfur, the TS value is raised by 50% compared to 5% SBS-only blend due to the vulcanizing action of the sulfur within the SBS polymer chain.

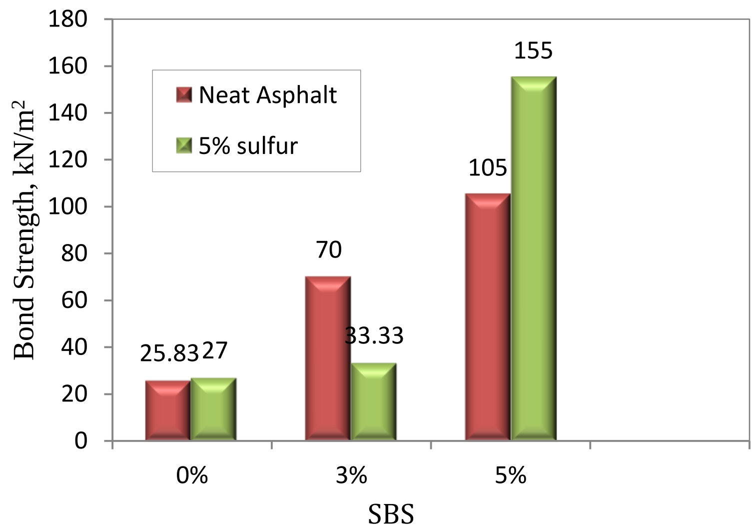

Figure 4-79: Bond strength of SBS-Sulfur mastic

RESULT SUMMARY

All the fillers (HOFA, CKD and LMD) causes increase in softening point and viscosity value of the asphalt mastic, but results in materials with less penetration and ductility than the neat asphalt, with HOFA having the greatest impact. The fillers have little or no effect on the flash point of the asphalt mastic.

Sulfur has negative effect on the flash point, softening point, viscosity and the ductility of the asphalt, but results in asphalt material with higher penetration at higher content (above 10% by weight of asphalt). This behavior of sulfur is maintained even when used in combination with the fillers, except at high (30% by weight of asphalt) content where there seem to be a little interaction between the filler grains and the sulfur crystals.

Oil sludge results in increased softening point, penetration and viscosity at doses below 10% by weight of asphalt, while it behaves in opposite manner at contents above 10%. It causes a continuous decline in ductility at all content range, and as with sulfur, sludge causes an obvious fall in flash point within 10% content, after which the FP decline rate reach a minimum. There seem to be interaction between the sludge and the fillers when combined, which lead to a reversal in behavior of the sludge additive towards the softening point and penetration.

Mod-EVA causes little increase in softening point and penetration of the asphalt, and produce mastics with lesser ductility and viscosity than the original asphalt. Mod-EVA results in tremendous decline in flash point value of the asphalt material, 5% by weight of asphalt is enough to bring down the FP by more than 120^{o}C. When mixed with the fillers, the Mod-EVA maintained its trend.

SBS causes significant rise in softening point and viscosity, while substantially reduced the penetration and ductility of the asphalt mastic. SBS has insignificant effect on the flash point unlike Mod-EVA. Mixing the SBS modified asphalt with fillers seem not improve it softening point and penetration, but causes more decrease in ductility and increase in viscosity.

CHAPTER 5

STATISTICAL ANALYSIS, PROPERTIES MODELS AND BLENDS POTENTIAL USE ASSESSMENT

Analysis of variance (ANOVA) was conducted using Minitab-16 statistical software application on all the properties result obtained with the additive as effects/factors [40]. This is to ascertain the relative effectiveness between the fillers within the extenders (sulfur, oil-sludge) and polymers, and also the extenders/polymers level of influence on the asphalt properties. Summary of the findings were given in the first coming sub-heading. The abstract of the ANOVA results were shown in *Table 5.1* through *Table 5.4*, but the complete Minitab print out is presented in *Appendix D.* And the detail description of the basic concept of ANOVA is also shown in *Appendix C.*

Properties regression models were discussed after the ANOVA result discussion, models for the various properties in terms of the percent additives content were summarized in *Table 5.5* through *Table 5.3*. The complete Minitab printout of the regression analysis can be found in *Appendix B.* Then the mastics were finally assessed in accordance with ASTM D449 and ASTM D312 for their potential use for roofing and waterproofing application.

5.1 RESULTS ANALYSIS OF VARIANCE (ANOVA)

Both HOFA and sulfur significantly affect the softening point (SP) for sulfur-filler asphalt blends except the other 2 fillers LMD and CKD. Oil-sludge in its self does not have any significant influence on the softening point when used along with HOFA and CKD fillers, unlike with LMD as can be seen from *Table 5.1*. But the fillers cause more significant rise on the SP value when paired with the sludge than when utilized alone, in other words, the little loss in SP due to the use of sludge can be compensated through the addition of CKD or HOFA. All the fillers (HOFA, CKD and LMD) show little effectiveness on improving the SP when used along with SBS polymer, the SBS causes a substantial rise in the SP to the extent that the filler end up reducing it or not affecting it. But SP is affected significantly by filler and Mod-EVA alike within Mod-EVA-filler asphalt mastics.

All participating additives in the sulfur-filler mastics caused a profound influence on the ductility of the asphalt material. Additional oil-sludge has little consequences on the ductility of the asphalt on top of the initial tremendous dent imposed by the first 20%, but it strengthen and increase the effectiveness of the fillers. Addition of the filler to the SBS asphalt blend cause a slight and insignificant change in its ductility due to the fact that more than 90% of the ductile property has been knocked out by the first 5% SBS dosage. But both Mod-EVA and filler actively participate in bringing down the ductility of Mod-EVA-filler blends, these can all be seen the ANOVA output summarized in *Table 5.2*.

Apart from HOFA filler all other additives have slight influence on the penetration of the sulfur-filler mastics. Individually oil-sludge significantly affects the penetration when used along with most of the fillers, but CKD in particular shows insignificant influence on the penetration compared to the rest of the filler. And as with the softening point property, the filler causes slight if not zero change upon the massive decrease resulting from the addition of the SBS polymer for SBS-filler mastics. And also the Mod-EVA as well as the fillers results in significant change in the penetration, all the aforementioned point and more can be deduced from *Table 5.3*.

Except for CKD and LMD, all other additives (sulfur and filler) significantly affect the viscosity of sulfur-filler mix. While the oil-sludge shows negligible effect on the viscosity as opposed to the filler additives which showed considerable rise in the viscosity of the sludge-filler blends. Like in all the rest of the properties, both the Mod-EVA and fillers actively result in significant change in the Mod-EVA-filler blends, for a complete picture of the above mentioned observations see *Table 5.4.*

Table 5.1: Result of Softening point ANOVA at 5% significance level

	SOFTENING POINT (^oC)				
	Factors/Additives	Tabular F_{value}	Calculated F_{value}	P-value	Comment
Sulfur – Filler blends	Sulfur	3.4903	8.67	0.002	Significant
	HOFA	3.2592	25.64	0.000	Significant
	Sulfur	3.4903	4.44	0.026	Significant
	CKD	3.2592	1.87	0.181	Insignificant
	Sulfur	3.4903	13.33	0.000	Significant
	LMD	3.2592	1.01	0.440	Insignificant
Sludge – Filler blends	Oil Sludge	4.4590	0.32	0.736	Insignificant
	HOFA	3.8379	6.96	0.010	significant
	Oil Sludge	4.4590	0.24	0.795	Insignificant
	CKD	3.8379	15.76	0.001	Significant
	Oil Sludge	4.4590	61.99	0.000	Significant
	LMD	3.8379	10.07	0.003	Significant
SBS - Filler blends	SBS	4.4590	54.08	0.000	Significant
	HOFA	3.8379	0.70	0.615	Insignificant
	SBS	4.4590	144.81	0.000	Significant
	CKD	3.8379	1.87	0.209	Insignificant
	SBS	4.4590	105.83	0.000	Significant
	LMD	3.8379	0.75	0.585	Insignificant
Mod-EVA–Filler blends	Mod-EVA	4.4590	1.24	0.338	Insignificant
	HOFA	3.8379	18.40	0.000	Significant
	Mod-EVA	4.4590	52.50	0.000	Significant
	CKD	3.8379	12.66	0.002	Significant
	Mod-EVA	4.4590	12.21	0.004	Significant
	LMD	3.8379	8.54	0.005	Significant

Table 5.2: Result of Ductility ANOVA at 5% significance level

	DUCTILITY (cm)				
	Factors/Additives	Tabular F_{value}	Calculated F_{value}	P-value	Comment
Sulfur – Filler blends	Sulfur	3.4903	0.33	0.803	Insignificant
	HOFA	3.2592	11.90	0.000	Significant
	Sulfur	3.4903	6.20	0.009	Significant
	CKD	3.2592	15.63	0.000	Significant
	Sulfur	3.4903	4.26	0.029	Significant
	LMD	3.2592	4.76	0.016	Significant
Sludge – Filler blends	Oil Sludge	4.4590	1.72	0.239	Insignificant
	HOFA	3.8379	2.82	0.099	Insignificant
	Oil Sludge	4.4590	5.84	0.027	Significant
	CKD	3.8379	2.17	0.163	Insignificant
	Oil Sludge	4.4590	7.96	0.013	Significant
	LMD	3.8379	1.30	0.349	Insignificant
SBS - Filler blends	SBS	4.4590	2.35	0.158	Insignificant
	HOFA	3.8379	1.35	0.332	Insignificant
	SBS	4.4590	8.69	0.010	Significant
	CKD	3.8379	1.27	0.356	Insignificant
	SBS	4.4590	11.81	0.004	Significant
	LMD	3.8379	1.28	0.353	Insignificant
Mod-EVA–Filler blends	Mod-EVA	4.4590	1.16	0.360	Insignificant
	HOFA	3.8379	4.07	0.044	Significant
	Mod-EVA	4.4590	9.40	0.008	Significant
	CKD	3.8379	6.13	0.015	Significant
	Mod-EVA	4.4590	9.48	0.008	Significant
	LMD	3.8379	3.04	0.085	Insignificant

Table 5.3: Results of Penetration ANOVA at 5% significance level

	PENETRATION (dmm)				
	Factors/Additives	Tabular F_{value}	Calculated F_{value}	P-value	Comment
Sulfur – Filler blends	Sulfur	3.4903	3.47	0.051	Insignificant
	HOFA	3.2592	7.76	0.003	Significant
	Sulfur	3.4903	9.41	0.002	Significant
	CKD	3.2592	0.12	0.972	Insignificant
	Sulfur	3.4903	10.56	0.001	Significant
	LMD	3.2592	0.62	0.659	Insignificant
Sludge – Filler blends	Oil Sludge	4.4590	0.24	0.795	Insignificant
	HOFA	3.8379	15.76	0.001	Significant
	Oil Sludge	4.4590	16.99	0.001	Significant
	CKD	3.8379	1.40	0.316	Insignificant
	Oil Sludge	4.4590	98.57	0.000	Significant
	LMD	3.8379	4.44	0.035	Significant
SBS - Filler blends	SBS	4.4590	1.64	0.253	Insignificant
	HOFA	3.8379	1.28	0.353	Insignificant
	SBS	4.4590	16.41	0.001	Significant
	CKD	3.8379	0.69	0.62	Insignificant
	SBS	4.4590	11.84	0.004	Significant
	LMD	3.8379	1.18	0.387	Insignificant
Mod-EVA–Filler blends	Mod-EVA	4.4590	12.36	0.004	Significant
	HOFA	3.8379	54.14	0.000	Significant
	Mod-EVA	4.4590	12.29	0.004	Significant
	CKD	3.8379	13.99	0.001	Significant
	Mod-EVA	4.4590	7.56	0.014	Significant
	LMD	3.8379	19.98	0.000	Significant

Table 5.4: Results of Viscosity ANOVA at 5% significance level

	Viscosity (cP)				
	Factors/Additives	Tabular F_{value}	Calculated F_{value}	P-value	Comment
Sulfur – Filler blends	Sulfur	3.4903	13.79	0.000	Significant
	HOFA	3.2592	72.85	0.000	Significant
	Sulfur	3.4903	43.61	0.000	Significant
	CKD	3.2592	3.12	0.056	Insignificant
	Sulfur	3.4903	55.57	0.000	Significant
	LMD	3.2592	1.91	0.173	Insignificant
Sludge – Filler blends	Oil Sludge	4.4590	3.34	0.088	Insignificant
	HOFA	3.8379	11.54	0.002	Significant
	Oil Sludge	4.4590	6.56	0.021	Significant
	CKD	3.8379	22.92	0.000	Significant
	Oil Sludge	4.4590	3.66	0.074	Insignificant
	LMD	3.8379	37.03	0.000	Significant
SBS - Filler blends	SBS	4.4590	**	**	**
	HOFA	3.8379	**	**	**
	SBS	4.4590	**	**	**
	CKD	3.8379	**	**	**
	SBS	4.4590	**	**	**
	LMD	3.8379	**	**	**
Mod-EVA–Filler blends	Mod-EVA	4.4590	8.81	0.009	Significant
	HOFA	3.8379	57.52	0.000	Significant
	Mod-EVA	4.4590	22.34	0.001	Significant
	CKD	3.8379	7.27	0.009	Significant
	Mod-EVA	4.4590	36.98	0.000	Significant
	LMD	3.8379	12.69	0.002	Significant

5.2 PROPERTIES REGRESSION MODELS

Minitab statistical software has been used to generate regression models of the various physical properties of asphalt mastic as a function of modifying additives. Excellent correlation between the predicted and actual response were mostly obtained with most of the coefficient of correlation (R^2) as high as 95%. The models were derived from combination of filler-sulfur within the ranges 0-25% and 0-30% respectively, filler-sludge within 0-25% and 0-40% ranges respectively and filler-polymer blends within 0-25% and 0-10% bounds respectively.

Most of the response surfaces turned out not to be linear plane, as a result majority of the models requires a supplementary quadratic terms. The variables should be substituted in decimal form as the models were generated using decimal inputs, for example 10% as 0.10; 15% as 0.15 and so on. And also all percent composition is by weight, the sulfur, polymer and sludge contents were expressed as the percentage of the asphalt weight and the filler content is expressed as the percentage of the total sulfur-asphalt, sludge-asphalt or polymer-asphalt mixture.

P-value of each variable within a particular model has been indicated, the regression analysis was run at 5% significance level, in other words at 5% allowance for possible error in the data. Variables with P-values less 5% are then said to be significant terms. Almost for all models only a few variable will be seen to be insignificant. Alphabets H, C, L, S, G, V and B were used to represent percentage composition of HOFA, CKD, LMD, Sulfur, Oil-Sludge, sulfur modified Eva and SBS polymer respectively.

5.2.1 SOFTENING POINT

Table 5.5: Regression models equations for Softening point

Additives	Regression models
Sulfur-HOFA HOFA = H Sulfur = S	**Softening point** = 52.1793 - 20.7***S + ** 56.7905***H** P-value (S) = 0.000 P-value (H) = 0.000 **R-Sq = 86.35%**
Sulfur-CKD CKD = C Sulfur = S	**Softening point** = 54.351 - 63.3657***S** + 96.2548***C*S** + 138.5***S**^{2} P-value (S) = 0.000 P-value (C*S) = 0.003 P-value(S^2) = 0.006 **R-Sq = 63.30%**
Sulfur-LMD LMD = L Sulfur = S	**Softening point** = 55.1465 - 83.0811**S**+5.41081**L**+35.8649**L*S**+189**S**^{2} P-value (S) = 0.000 P-value (L*S) = 0.477 P-value(S^2) = 0.001 P-value (L) = 0.565 **R-Sq = 75.37%**
Sludge-HOFA HOFA = H Sludge = G	**Softening point** = 53.5432 - 21.8514**G** + 53.7387**H** + 186.081**G*H** P-value (G) = 0.007 P-value (H) = 0.000 P-value (G*H) = 0.001 **R-Sq = 95.21%**
Sludge-CKD CKD = C Sludge = G	**Softening point =** 51.9125 - 11.2973**G** + 33.5733**C** + 77.8378**G*C** - 81.7084**C**^{2} P-value (G) = 0.014 P-value (C) = 0.030 P-value (G*C) = 0.007 P-value (C^2) = 0.121 **R-Sq = 87.92%**
Sludge-LMD LMD = L Sludge = G	**Softening point =** 53.0103 - 17.2***G** + 19.1171***L** P-value (G) = 0.000 P-value (L) = 0.000 **R-Sq = 90.59%**
Mod-Eva-HOFA HOFA = H Mod-Eva = V	**Softening point =** 53.4184 + 9.2**V** + 51.2973**H** P-value (V) = 0.495 P-value (H) = 0.000 **R-Sq = 85.15%**
Mod-Eva-CKD CKD = C Mod-Eva = V	**Softening point =** 51.839 + 67.4**V** + 20.4595**C** P-value (V) = 0.000 P-value (H) = 0.000 **R-Sq = 90.41%**
Mod-Eva-LMD LMD = L Mod-Eva = V	**Softening point =** 53.4656 - 47.6**V** + 19.5315**L** + 752**V**^{2} P-value (V) = 0.060 P-value (L) = 0.000 P-value (V^2) = 0.005 **R-Sq = 87.03%**

For the original models complete Minitab printout and SBS blend models check

appendix-B

5.2.2 DUCTILITY

Table 5.6: Regression models for ductility

Additives	Regression models
Sulfur-HOFA HOFA = H Sulfur = S	**Ductility (cm) =**122.644 -191.178**S -** 866.764**H+**1083.7**H*S+**1518.02**H**^{2} P-value (S) = 0.004 P-value (H) = 0.000 P-value (H*S) = 0.007 P-value (H^2) = 0.008 **R-Sq = 86.20%**
Sulfur-CKD CKD = C Sulfur = S	**Ductility** (cm) =135.225-259.959**S**-761.723**C**+947.568S*****C**+1358.76 **C**^{2} P-value (S) = 0.000 P-value (C) = 0.000 P-value (C*S) = 0.000 P-value (C^2) = 0.000 **R-Sq = 95.01%**
Sulfur-LMD LMD = L Sulfur = S	**Ductility** (cm) = 139.721 - 286.819**S**- 383.249**L** + 1159.14 **L*S** P-value (S) = 0.000 P-value (L) = 0.000 P-value (L*S) = 0.000 **R-Sq = 85.99%**
Sludge-HOFA HOFA = H Sludge = G	**Ductility =** 118.57-348.484**G**-811.198**H**+1014.53**G*H**+329.25**G**^{2}+1400.64**H**^{2} P-value (G) = 0.022 P-value (H) = 0.004 P-value (G*H) = 0.023 P-value (G^2) = 0.262 P-value (H^2) = 0.101 **R-Sq = 80.45%**
Sludge-CKD CKD = C Sludge = G	**Ductility =** 126.822-418.681**G**-621.452**C**+927.365**G*C**+425.25**G**^{2}+ 980.795**C**^{2} P-value (G) = 0.002 P-value (C) = 0.003 P-value (**G*C**) = 0.008 P-value (G^2) = 0.068 P-value (C^2) = 0.119 **R-Sq = 87.45%**
Sludge- LMD LMD = L Sludge = G	**Ductility (cm) =** 135.188-504.07**G**-376.342**L**+1066.22**G*L**+534.5**G**^{2} P-value (G) = 0.000 P-value (L) = 0.000 P-value (G*L) = 0.001 P-value (G^2) = 0.010 **R-Sq = 80.45%**
Mod-Eva- HOFA HOFA = H Mod-Eva = V	**Ductility (cm) =** 125.729 - 835.486**V** - 875.356**H** + 4169.19**V*H** + 1520.17**H**^{2} P-value (V) = 0.040 P-value (H) = 0.002 P-value (V*H) = 0.001 P-value (H^2) = 0.007 **R-Sq = 84.80%**
Mod-Eva- CKD CKD = C Mod-Eva = V	**Ductility (cm) =** 133.677 - 987.622**V** - 804.793**C** + 3417.3**V*C** + 1564.21**C**^{2} P-value (V) = 0.000 P-value (C) = 0.000 P-value (V*C) = 0.002 P-value (C^2) = 0.004 **R-Sq = 93.25%**
Mod-Eva- LMD LMD = L Mod-Eva = V	**Ductility (cm) =** 139.729 - 1128.3**V** - 414.09**L** + 3997.84**V*L** P-value (V) = 0.000 P-value (L) = 0.000 P-value (V*L) = 0.000 **R-Sq = 96.52%**

For the original models complete Minitab printout and SBS blend models check appendix-B

5.2.3 PENETRATION

Table 5.7: Penetration results regression models

Additives	Regression models
Sulfur-HOFA HOFA = H Sulfur = S	**Penetration** (dmm) = 55.1806 + 139.27 **S**-257.814**H** + 580.015 $\mathbf{H^2}$ - 309.5 $\mathbf{S^2}$ P-value (S) = 0.022 P-value (H) = 0.002 P-value (S^2) = 0.097 P-value (H^2) = 0.042 **R-Sq = 76.63%**
Sulfur-CKD CKD = C Sulfur = S	**Penetration** (dmm) = 47.796 + 369.96 **S** + 3.02703**C**- 1030 $\mathbf{S^2}$ P-value (S) = 0.000 P-value (0.902) = 0.000 P-value (S^2) = 0.000 **R-Sq = 68.73%**
Sulfur-LMD LMD = L Sulfur = S	**Penetration** (dmm) = 53.517+166.615**S**+239.393**S*L** - 249.406$\mathbf{L^2}$ - 394.5$\mathbf{S^2}$ P-value (S) = 0.005 P-value (L*S) = 0.116 P-value (S^2) = 0.018 P-value (L^2) = 0.030 **R-Sq = 77.10%**
Sludge-HOFA HOFA = H Sludge = G	**Penetration** = 53.8395-21.8514**G**+151.748**H**+186.081**G*H**-1265.66$\mathbf{H^2}$+ 3599.16$\mathbf{H^3}$ P-value (G) = 0.001 P-value (H) = 0.005 P-value (G*H) = 0.000 P-value (H^2) = 0.016 P-value (H^3) = 0.010 **R-Sq = 98.10%**
Sludge-CKD CKD = C Sludge = G	**Penetration** (dmm) = 54.2624+416.073**G**-42.4459**C**-220.878**G*C**-772.75$\mathbf{G^2}$ P-value (G) = 0.000 P-value (C) = 0.438 P-value (G*C) = 0.303 P-value (G^2) = 0.000 **R-Sq = 84.09%**
Sludge- LMD LMD = L Sludge = G	**Penetration** (dmm) = 58.9604 + 159.5 **G** - 86.2883**L** P-value (G) = 0.000 P-value (L) = 0.001 **R-Sq = 95.55%**
Mod-Eva-HOFA HOFA = H Mod-Eva = V	**Penetration** (dmm**)** = 64.4919 + 108.8**V** - 333.588**H** + 674.953$\mathbf{H^2}$ P-value (V) = 0.006 P-value (H) = 0.000 P-value (H^2) = 0.005 **R-Sq = 93.13%**
Mod-Eva-CKD CKD = C Mod-Eva = V	**Penetration** (dmm) = 64.7819 + 532.2**V** - 167.405**C** – 5124$\mathbf{V^2}$ + 260.55$\mathbf{C^2}$ P-value (V) = 0.001 P-value (C) = 0.005 P-value (G*C) = 0.001 P-value (V^2) = 0.174 **R-Sq = 89.14%**
Mod-Eva-LMD LMD = L Mod-Eva = V	**Penetration** (dmm) = 67.563 + 53.108**V** - 276.193**L** + 323.514**V*L** + 670.29$\mathbf{L^2}$ P-value (V) = 0.268 P-value (L) = 0.000 P-value (V*L) = 0.267 P-value (L^2) = 0.001 **R-Sq = 91.83%**

For the original models complete Minitab printout and SBS blend models check appendix-B

5.2.4 VISCOSITY

Table 5.8: Viscosity results penetration models

Additives	Regression models
Sulfur-HOFA HOFA = H Sulfur = S	**viscosity** (cP) =680.75 - 1263.2**S** - 3008.94**H**-14477.8**S*H** + 64590.4 $\mathbf{H^2}$ P-value (S) = 0.318 P-value (H) = 0.323 P-value (S*H) = 0.070 P-value (H^2) = 0.000 **R-Sq = 93.96%**
Sulfur-CKD CKD = C Sulfur = S	**viscosity** (cP) = 677.403 - 3325**S** + 614.562**C** - 4125**S*C**+2918.81$\mathbf{C^2}$ + 7125$\mathbf{S^2}$ P-value (S) = 0.000 P-value (C) = 0.284 P-value (C*S) = 0.010 P-value (C^2) = 0.162 P-value (S^2) = 0.000 **R-Sq = 95.20%**
Sulfur-LMD LMD = L Sulfur = S	**viscosity** (cP) = 703.581 - 3558.45**S** + 810.135**L** - 2118.24**S*****L**+7250$\mathbf{S^2}$ P-value (S) = 0.000 P-value (L) = 0.007 P-value (S*L) = 0.148 P-value (S^2) = 0.000 **R-Sq = 93.96%**
Sludge-HOFA HOFA = H Sludge = G	**viscosity** (cP) = 1422.91-3599.73**G**-29439.2**H**+71608.8**G*H**+177136$\mathbf{H^2}$ P-value (G) = 0.195 P-value (H) = 0.009 P-value (G*H) = 0.001 P-value (H^2) = 0.000 **R-Sq = 95.10%**
Sludge-CKD CKD = C Sludge = G	**viscosity** (cP) = 581.863 - 99.6284**G** + 1713.24**C** + 4102.7**G*C** P-value (G) = 0.563 P-value (C) = 0.000 P-value (G*C) = 0.002 **R-Sq = 96.20%**
Sludge- LMD LMD = L Sludge = G	**viscosity** (cP) = 625.013-298.142**G**+1796.34**L**+1013.51**G*L**-1989.49$\mathbf{L^2}$ P-value (G) = 0.053 P-value (L) = 0.003 P-value (G*L) = 0.248 P-value (L^2) = 0.272 **R-Sq = 93.01%**
Mod-Eva-HOFA HOFA = H Mod-Eva = V	**viscosity** (cP) = 790.986 + 606.081**V** - 8606.4**H** + 76035.1**V*H** + 91530.6$\mathbf{H^2}$ P-value (V) = 0.885 P-value (H) = 0.036 P-value (V*H) = 0.0012 P-value (H^2) = 0.000 **R-Sq = 97.50%**
Mod-Eva-CKD CKD = C Mod-Eva = V	**viscosity** (cP) = 645.529 - 2196.97**V** + 1406.94**C** + 36378.4**V*C** + 54040$\mathbf{V^2}$ P-value (V) = 0.350 P-value (C) = 0.008 P-value (V*C) = 0.000 P-value (V^2) = 0.020 **R-Sq = 97.54%**
Mod-Eva-LMD LMD = L Mod-Eva = V	**viscosity** (cP) = 631.721 + 2702.7**V** + 1136.76**L** + 15337.8 **V*L** P-value (V) = 0.006 P-value (L) = 0.004 P-value (V*****L) = 0.008 **R-Sq = 95.69%**

For the original models complete Minitab printout and SBS blend models check appendix-B

5.3 BLENDS POTENTIAL AND SUITABLE AREA OF APPLICATION ASSESSMENT

The various asphalt mastics were assessed in accordance to ASTM D449: *Asphalt used in Damp-proofing and waterproofing specification*, and ASTM D332: *Standard specification for asphalt used in roofing.* The first specification (ASTM D449) covers three types of asphalt suitable for use as a mopping coat in damp proofing; or as a plying or mopping cement in the construction of membrane waterproofing systems with felts, fabrics, and asphalt-impregnated glass mat. While ASTM D332 covers four types of asphalt intended for use in built-up roof construction, construction of some modified bitumen systems, construction of bituminous vapor retarder systems, and for adhering insulation boards used in various types of roof systems.

5.3.1 ASTM D 449: *Asphalt used in Damp-proofing and waterproofing specification*

Classification:

Type I – Soft, adhesive, "self-healing" asphalts that flows easily under the mop and is suitable for use below grade under uniformly moderate temperature condition both during the process of installation and during service. It is suitable for foundations, tunnels, subways, etc.

Type II – A somewhat less susceptible asphalt than Type I, with good adhesive and "self healing" properties, suitable for use above grade where it will not be exposed to temperatures exceeding 50^{o}C. Type II is suitable for railroad bridges, culverts, retaining walls, tanks, dams, conduits, spray decks etc

Table 5.9: Damp-proofing and waterproofing material classification

Asphalt		Heavy oil fly ash 'HOFA'					Cement kiln dust 'CKD'				Lime Dust 'LD'			
		0%	10%	15%	20%	25%	10%	15%	20%	25%	10%	15%	20%	25%
	0%	I	II	II	II	II	I	I	I	I	I	I	I	I
Sulfur	10%	X	X	X	X	X	X	X	X	X	X	X	X	X
	20%	X	X	X	X	X	X	X	X	X	X	X	X	X
	30%	X	X	X	X	X	X	X	X	X	X	X	X	X
Mod-EVA	5%	X	X	X	X	X	X	X	X	X	X	X	X	X
	10%	X	X	X	X	X	X	X	X	X	X	X	X	X
SBS	5%	III	III	III	III	X	III	III	III	III	III	III	III	III
	10%	X	III	III	X	X	III	III	III	III	III	III	III	III
Oil Sludge	20%	I	X	X	X	X	X	X	X	X	I	I	I	I
	40%	I	X	II	X	X	X	X	X	X	X	X	X	X

Type III – An asphalt less susceptible to temperature than type II, with good adhesive properties, and suitable for use above grade on vertical surface exposed to direct sunlight or temperatures above 50^{o}C.

Type X – Any asphalt mastic that fails to meet the specification requirements in terms of lower softening point, ductility or flash point prescribed level. Where applicable, the mastic should be used within the limits determined by physical tests.

Table 5.9 shows the category to which each composite belong. Most of the sludge-HOFA mixes should have fall under Type II class, If not for the short fall in their ductile property. The CKD-sludge mixes are supposed to under type I material, but they are deficient in flash value, this relegates them to a position outside the class range type. The 40% sludge-LMD materials which should also have been type I like the 20% sludge-LMD blends could not pass the ductility and some even the penetration standards. The SBS composites are all type III materials if not for some few that fail to meet the ductility condition. On the other hand all modified-EVA mixes fail to satisfy the flash point condition with some even additionally violating the ductility requirement, so cannot be classified under any type/group. All the sulfur containing blend also fail to meet the minimum Flash point set-level, in addition to this some also the minimum ductility, if not they all might have been classified under type I material. But the HOFA-only mixes are all type II, and all the CKD and LMD-only blends fall under type I category.

5.3.2 ASTM D 312: *Standard specification for asphalt used in roofing*

Classification:

Classification is based on four suggested slope guidelines for the four types of asphalts based on the slope at which specific asphalt will be used. However, no restriction are implied or intended on the slope at which a specific type of asphalt must be used.

Type I: Includes asphalt relatively susceptible to temperature, with good adhesive and self healing properties for use in smooth and slag- or gravel surfaced roofing on inclines generally not exceeding 1 in/ft.

Type II**:** includes asphalts moderately susceptible to temperature for roofing laid on inclined generally greater than 1in. up to and including 3 in./ft. and may be either smooth or surfaced with slag or gravel.

Type III: includes asphalts relatively not susceptible to temperature for use on inclines generally in the range of more than 50%.

Type IV: includes asphalt that are generally not susceptible to flow at room temperature for use in construction of built-up roofing on inclines from approximately 16.7% (2in./ft.) slope to 50% (6in./ft) slope. These asphalts may be useful in areas where relatively high year round temperatures are experienced.

Type X – Any asphalt mastic that fails to meet the specification requirements in terms of lower softening point, ductility or flash point prescribed level. Where applicable, the mastic should be used within the limits determined by physical tests.

According to ASTM D 312 (Standard specification of asphalt used in roofing) majority of the sulfur-asphalt blends did not pass the minimum flash point requirement to be classified for roofing application. In addition to this some did not also pass the minimum softening point criteria. But the HOFA-only blends fall under Type I, only 25% HOFA composite scaled to be under Type II. However none of the LMD- and CKD-only blends pass the minimum softening point value, if not for 20% and 25% LMD mixes which barely manage to scale and fall Under Type I.

The neat asphalt cannot be classed under ASTM D312, because it does not meet minimum SP mark, but when modified as can be seen from *Table 5.11*, the resulting composite can be classified for roofing application according to the specification. Even though the modified-EVA blends possessed sufficient SP to be within the classes range, none was able to meet the FP requirements. As for SBS mixes, majority come under Type III material with few moving up to Type IV category. The sludge-only blends can be classified under type I material, but the double-additive blends containing LMD and CKD in addition to sludge did not pass the SP criteria and all fail to satisfy the FP minimum set-value. Table 5.10 shows a recommended ranges of additive content for the two category of applications, based on the previous assessment.

Table 5.10: Recommended mastic additive content for various application

	Recommended additive composition		
	Roofing application		
	Sulfur/Sludge	HOFA	LMD/CKD
Sulfur-Filler mastic	0 - 30%	15% - 25%	15% - 35%
Sludge-filler mastic	0 - 40%	10% - 25%	15% - 35%
Waterproofing and Damp proofing application			
Sulfur-Filler mastic	0 - 30%	0% - 25%	0% - 25%
Sludge-filler mastic	0 - 40%	0% - 15%	0% - 25%

Table 5.11: Roofing application material classification of mixes

Asphalt		Heavy oil fly ash 'HOFA'					Cement kiln dust 'CKD'				Lime Dust 'LD'			
		0%	10%	15%	20%	25%	10%	15%	20%	25%	10%	15%	20%	25%
	0%	X	I	I	I	II	X	X	X	X	X	X	I	I
Sulfur	10%	X	X	X	X	X	X	X	X	X	X	X	X	X
	20%	X	X	X	X	X	X	X	X	X	X	X	X	X
	30%	X	X	X	X	X	X	X	X	X	X	X	X	X
Mod-EVA	5%	X	X	X	X	X	X	X	X	X	X	X	X	X
	10%	X	X	X	X	X	X	X	X	X	X	X	X	X
SBS	5%	III	III	III	X	X	II	II	III	III	II	II	III	III
	10%	IV	III	III	X	X	III	III	IV	IV	III	III	III	III
Oil Sludge	20%	X	I	X	X	X	X	X	X	X	X	X	X	X
	40%	X	I	X	X	X	X	X	X	X	X	X	X	X

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

The summary of the general findings from the results analysis were presented in the subsequent sub-heading, test-wise. Based on the graphical and statistical interpretation of the data obtained from the various test conducted, the trend and manner in which the different additives (HOFA, CKD, Sulfur, LMD, SBS, sulfur modified EVA and Sludge) affect each property were highlighted.

6.1.1 Softening Point (SP)

1. Generally the softening point is negatively affected with more sulfur additive and positively influenced with increasing HOFA. An increase in softening point is also observed with higher lime dust (LMD) content, but unlike HOFA, the LMD has little influence on this parameter. But In contrast to HOFA and LMD, the effect of CKD on softening point can be observed to be uniform for CKD-only mixes with $1^{o}C$ rise for every 5% content.

2. The softening point (SP) drops with increase in the amount of oil-sludge (OS) for doses above 10% by weight of the asphalt, below this quantity it seems to be rising.
3. The softening point ‘SP’ tend to rise uniformly with increase in mod-EVA content as expected, an average of $2^{o}C$ increment is recorded for every 5% addition of mod-EVA.
4. Styrene Butadiene Styrene ‘SBS’ has a tremendous effect on the softening point (SP) of the asphalt. The initial 5% SBS content yield material with an SP increase of about $34^{o}C$ compared to the neat asphalt.

6.1.2 Ductility

1. Addition of sulfur to the asphalt results in low ductile composite, a loss of more than 50% in ductility can be observed at 20% sulfur content. Even though both sulfur and HOFA negatively affect the ductility individually, a material with relatively higher ductility than purely HOFA-blend is obtained when they are combined. The ductility also decreases with more lime dust, but not as considerably as in the case of HOFA. And CKD-asphalt blend shows lower ductile behavior compared to their LMD blend counter parts.
2. Oil sludge (OS) has a negative effect on the ductility of the asphalt material.
3. Modified-EVA ‘mod-EVA’ results in material blend with lower ductility, 5% of Mod-Eva yield composite having 50% less ductile property relative to the original asphalt, and the 10% content causes about 75% loss in ductility.

4. Less ductile materials results from the addition of Styrene Butadiene Styrene 'SBS' to the asphalt, 10% SBS is sufficient to almost eliminate the ductile property of the original asphalt completely.

6.1.3 Penetration

1. At ranges between 0 – 10% sulfur the penetration seem to be declining with more sulfur, while beyond this interval it can be seen to be rising until it is higher than that of neat asphalt at 30%. The effectiveness of HOFA in cutting the penetration value of the asphalt has been attenuated by sulfur additive sulfur in the Sulfur-HOFA blends. Similar to LMD mix, the CKD blends have an increased penetration with lower CKD.
2. The oil sludge (OS) increase the penetration value of the asphalt composite at doses above 10%, an average uniform increase of about 20dmm for every 20% increase in OS can be observed for OS-only blends.
3. The penetration seems to be increasing with the addition of modified-EVA.
4. The styrene-butadiene-styrene 'SBS' causes a large decrease in penetration value of the asphalt by more than 50% for the first 5% SBS content.

6.1.4 Viscosity

1. The addition of sulfur to neat asphalt causes a gradual drop in its viscosity. On the other hand HOFA shows a tremendous thickening ability, which could be attributed to its ability to absorb the oily constituent of the asphalt that in turn result to higher interlayer friction. The limestone dust does not seem to change the

viscosity significantly, and the CKD blends more or less behave in similar manner as the LMD mixes.

2. The viscosity tend to be increasing with more sludge for doses below 10%, after which it starts declining until it falls a bit below the original asphalt viscosity at 40% OS content by 37.5 centi-poise.
3. The viscosity slightly increases with more modified-EVA ‘mod-EVA’.
4. The 10% SBS modified asphalt produce composite of high viscosity beyong experiment limit setup (20rpm, 135^{o}C) for the entire filler content range.

6.1.5 Flash Point (FP)

1. The mineral fillers and HOFA has little or no effect on the flash point, the maximum difference between neat asphalt value and the highest filler content (25%) is not more than 15^{o}C which is very little compared to that 340^{o}C. Similar trend as in the HOFA-Sulfur blend is observed for the LMD-Sulfur mixes. But the horizontal change in flash due to filler increase tend to be a little more pronounced with the CKD as compared to the other two additives (HOFA and LMD) especially for the CKD-only blends.
2. The oil-sludge (OS) has decreasing effect on the flash point (FP) of the asphalt, 20% content result to a decline of about 80^{o}C, but this effect becomes less pronounce with more additional OS.
3. As the case with sulfur and sludge, the modified-EVA ‘mod-EVA’ has tremendous negative effect on the flash point ‘FP’ of the asphalt material, which can be largely attributed to its sulfur constituent.

4. The SBS polymer has little or no effect on the flash point 'FP' of the asphalt material, both the 5% and 10% SBS modified asphalt shows FP value of 330^{o}C, which is more or less equal to that of the neat asphalt (340^{o}C).

6.1.6 Bond Strength (BS)

1. Asphalt bond strength (BS) increase with both sulfur and Oil sludge initially to a certain peak, then, it starts to decline at higher content.
2. The SBS causes a linear increment in the asphalt's tensile strength while Mod-EVA shows slight or no improvement in BS.
3. Addition of mineral filler (HOFA/CKD/LMD) to the asphalt generally results to an increase in BS. The SBS-modified asphalt exhibit almost twice the bond strength possessed by the 25% CKD and LMD containing asphalt at 5% SBS content.
4. At 5% SBS and 5% sulfur, the BS value is raised by 50% compared to 5% SBS-only blend due to the vulcanizing action of the sulfur within the SBS polymer chain.

6.1.7 ASTM SPECIFICATIONS

Most of the sludge blends meet the requirement of ASTM D449 and ASTM D312 specifications if not their lack of sufficient ductility. All the sulfur Mod-EVA mastics fail to meet the minimum flash point requirement of ASTM D449 and ASTM D312 specifications. All the SBS mastics meet the requirement of both specifications.

6.2 RECOMMENDATIONS

Based on the results obtained from this research work and the analysis provided, out of the numerous possible suggestion triggered by vivid observations, the following important recommendations are made:

1. When assessing the relative effect of two or more inert mineral fillers the percent composition should be expressed by volume not by weight, this will yield the most accurate comparative result as the influence of the molecular weight of the mineral particles will be eliminated through the use of volumetric indicator which provide uniform number of particles for all the fillers. Since the interaction between the asphalt and filler particulates is a physical one, the modifying ability of any filler will be more a function of the amount of its grain present than its mass.
2. The current penetration test is not a good indicator of hardness change in mineral-filled asphalt due to the fact that the thin penetrating needle head has the tendency of passing through even the most tight space between the asphalt and the filler particles, hence missing the overall effect of the filler grains especially when in minor quantities. A thin chisel-like needle head which can cover a sufficient linear space should be prescribed for testing filled asphalt.
3. Asphalt mastic bond strength test should be standardized and included in the standard test methods for preformed expansion joint filler for concrete construction (ASTM D545-08).
4. Further research on the vulcanizing effect of sulfur in polymer modified asphalt on the various properties of the roofing asphalt should be pursued, as the

phenomena has the potential of saving the necessity of using large amount polymer additive for different modifying effect.

5. Even though no specific additive content is monopolized for single application, the following range are recommended for the various categories of use:

   Roofing application

   - Sulfur-filler mastic should consist of up to 30% sulfur in combination with HOFA ranging from 15% to 25%, or LMD/CKD varying in content between 15% to 35%.
   - Sludge-filler mastics should contain up to 40% oil sludge in combination with 10% to 25% HOFA, or 15% to 35% LMD/CKD.

   Waterproofing and Damp proofing application:

   - Sulfur-filler mastic should consist of up to 30% sulfur in combination with HOFA ranging from 0% to 25%, or LMD/CKD varying in content between 0% to 25%.
   - Sludge-filler mastics should contain up to 40% oil sludge in combination with 0% to 15% HOFA, or 0% to 25% LMD/CKD.

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APPENDIX A

DISCRETE TABULAR PROPERTIES DATA

Table A 15: R & D softening point results for Heavy oil Fly Ash (HOFA) mixes

Temp (^{o}C)	0%(HOFA)	10%(HOFA)	15%(HOFA)	20%(HOFA)	25%(HOFA)
0%sulfur	52.3	61.8	62.2	61.2	68.1
10% sulfur	52.9	53.7	54.8	59.2	61.9
20% sulfur	48.5	53.2	54.6	60.3	64.1
30% sulfur	45.1	50.4	53.8	58.2	64.2
5%mod.Eva	54.1	55.3	59.0	64.0	67.7
10%mod.Eva	56.7	59.6	62.1	63.7	68.1
5% SBS	86.9	85.0	84.5	92.1	95.0
10% SBS	106.9	92.5	93.3	95.0	96.6
20% Sludge	49.8	58.1	61.7	65.9	73.5
40% Sludge	46.4	57.1	62.4	67.8	80.3
20%Sulfur + 5%mod.Eva	53.5	61	62.6	64.7	69.9
30%Sulfur + 5%mod.Eva	52.9	61	64	67.6	71.2

Table A 2: R & D softening point results for Limestone Dust (LMD) mixes

Temp (^oC)	0% (LMD)	10% (LMD)	15% (LMD)	20% (LMD)	25% (LMD)
0%sulfur	52.3	56.2	56.3	57.2	59
10% sulfur	52.9	47.5	48.8	48.4	47.9
20% sulfur	48.5	47.3	48.5	49.2	50.2
30% sulfur	45.1	48.4	49.1	51.3	52.1
5%mod.Eva	54.1	54	54.2	57.3	58.9
10%mod.Eva	56.7	58.4	60	59.3	60.4
5% SBS	86.9	82.2	83.8	87.6	89.5
10% SBS	106.9	91.4	92.7	93.4	94.0
20% Sludge	49.8	50.5	51.7	52.3	51.8
40% Sludge	46.4	47.5	49.9	51.3	51.5
20%Sulfur + 5%mod.Eva	53.5	57.4	56.7	57.6	57.6
30%Sulfur + 5%mod.Eva	52.9	57.4	57.4	58.8	59.4

Table A 3: R & D softening point results for Cement Kiln Dust (CKD) mixes

Temp (^oC)	0% (CKD)	10% (CKD)	15% (CKD)	20% (CKD)	25% (CKD)
0%sulfur	52.3	53.6	55.1	56.1	56.3
10% sulfur	52.9	47.1	48.4	50.6	49.8
20% sulfur	48.5	50.6	51	52.1	52.3
30% sulfur	45.1	51.9	52.2	53.6	54.8
5%mod.Eva	54.1	59.9	58.8	59	58.8
10%mod.Eva	56.7	61.7	61.8	63.4	63.5
5% SBS	86.9	82	83.4	85.6	87.1
10% SBS	106.9	88.9	90.7	100.3	101.2
20% Sludge	49.8	54.2	54.8	54.7	55.4
40% Sludge	46.4	55.1	55.3	55.9	59.9
20%Sulfur + 5%mod.Eva	53.5	56.9	57.2	59.8	59.4
30%Sulfur + 5%mod.Eva	52.9	59.7	59.1	60.1	59.9

Table A 4: Ductility at 25^{o}C (5cm/min) for Heavy oil fly ash (HOFA) blends

Ductility(cm)	0%(HOFA)	10%(HOFA)	15%(HOFA)	20%(HOFA)	25%(HOFA)
0%sulfur	150+	18.9	19.4	18.1	7.7
10% sulfur	104	29.3	24.2	23.5	8.9
20% sulfur	68	37.2	33.2	18.7	10.4
30% sulfur	63	33.4	30.2	23.7	5.5
5%mod.Eva	73	34	32.5	20.7	12
10%mod.Eva	34.5	22.2	13	13	5.5
5% SBS	14.5	5.2	3.5	1.5	1
10% SBS	4	2.8	2.3	1.5	1.3
20% Sludge	35	21	9.5	5.5	2.5
40% Sludge	31	18.3	10.5	3.5	1.3
20%Sulfur + 5%mod.Eva	51.5	9.5	6.5	3.5	1.5
30%Sulfur + 5%mod.Eva	44.8	11	4	2.5	1.5

Table A 5: Ductility at 25^{o}C (5cm/min) for Limestone dust (LMD) blends

Ductility(cm)	0% (LMD)	10% (LMD)	15% (LMD)	20% (LMD)	25% (LMD)
0%sulfur	150+	94.5	70.5	66.5	31
10% sulfur	104	106.4	67.5	65.5	64.3
20% sulfur	68	57	65.5	49.5	42
30% sulfur	63	47.5	57	42.5	36.5
5%mod.Eva	73	55.2	51.5	41	38.5
10%mod.Eva	34.5	25.8	23.4	23	21.5
5% SBS	14.5	48	34.5	7.5	4.5
10% SBS	4	5.5	7.5	6	4
20% Sludge	35	36.6	27.5	33	32.5
40% Sludge	31	27.5	22.5	22.5	27
20%Sulfur + 5%mod.Eva	51.5	20	20.5	21.3	20
30%Sulfur + 5%mod.Eva	44.8	20.8	19.3	19.2	20.9

Table A 6: Ductility at 25^{o}C (5cm/min) for Cement-Kiln-dust (CKD) blends

Ductility(cm)	0% (CKD)	10% (CKD)	15% (CKD)	20% (CKD)	25% (CKD)
0%sulfur	150+	57	49.5	40	35
10% sulfur	104	64	35.5	29.5	27
20% sulfur	68	48.5	25.5	24	23
30% sulfur	63	22.5	22.5	16	19
5%mod.Eva	73	20.5	20	26.5	18.3
10%mod.Eva	34.5	14.5	10.5	10	7.4
5% SBS	14.5	8	7	5.5	4.4
10% SBS	4	5.2	7.2	2.6	2.6
20% Sludge	35	29	21.7	21	21
40% Sludge	31	20.5	15.5	15.5	11.5
20%Sulfur + 5%mod.Eva	51.5	20.8	20	17.8	14.5
30%Sulfur + 5%mod.Eva	44.8	23	18.8	19.8	19

Table A 7: Penetration at 25^{o}C under 100g load for 5sec. for Heavy oil fly ash (HOFA) blends

Penetr.(dmm)	0%(HOFA)	10%(HOFA)	15%(HOFA)	20%(HOFA)	25%(HOFA)
0%sulfur	67.6	41.0	40.3	34.7	33.0
10% sulfur	55.9	45.8	44.1	40.1	33.7
20% sulfur	65.9	54.1	45.8	41.7	35.9
30% sulfur	70.9	55.6	53.4	46.1	40.6
5%mod.Eva	70	48.6	41.7	34.9	31.5
10%mod.Eva	73.9	46.8	38.8	33.8	30.9
5% SBS	24.1	33.7	31.9	25.2	23
10% SBS	21.6	29.2	24.2	23.5	19.4
20% Sludge	84.7	71.3	68.5	56.1	41.1
40% Sludge	111.8	45.8	40.3	34.7	55.9
20%Sulfur + 5%mod.Eva	85.5	64	56.4	46.2	40.9
30%Sulfur + 5%mod.Eva	85.2	48.2	42.2	36.6	30.3

Table A 8: Penetration at 25^{o}C under 100g load for 5sec. for Limestone dust (LMD) blends

Penetr.(dmm)	0% (LMD)	10% (LMD)	15% (LMD)	20% (LMD)	25% (LMD)
0%sulfur	67.6	46.1	44.5	37.7	35.9
10% sulfur	55.9	67.8	62.7	64.9	65.7
20% sulfur	65.9	73.4	72.5	70.8	67.1
30% sulfur	70.9	79.1	74.7	72.0	68.4
5%mod.Eva	70	47.6	43.8	50.7	53.2
10%mod.Eva	73.9	54.9	51.6	51.4	49.5
5% SBS	24.1	33.7	31.9	25.2	23
10% SBS	21.6	29.2	24.2	23.5	19.4
20% Sludge	84.7	86.8	82.4	74.3	70.9
40% Sludge	111.8	124.3	111.6	110.2	92.9
20%Sulfur + 5%mod.Eva	85.5	58.5	56.7	54.4	51.8
30%Sulfur + 5%mod.Eva	85.2	58.9	56.2	49	49.5

Table A 9: Penetration at 25oC under 100g load for 5sec. for Cement Kiln Dust (CKD) blends

Penetr.(dmm)	0% (CKD)	10% (CKD)	15% (CKD)	20% (CKD)	25% (CKD)
0%sulfur	67.6	46.9	44.5	43.8	38.8
10% sulfur	55.9	72.5	75.9	81.5	78.8
20% sulfur	65.9	86.8	92.6	82.6	82.9
30% sulfur	70.9	68.4	63.8	62.8	61.9
5%mod.Eva	70	64.9	63.9	60.1	52
10%mod.Eva	73.9	50	45	44.4	38.5
5% SBS	24.1	30.3	28.2	24.4	23.4
10% SBS	21.6	27.3	26.8	25.9	23.7
20% Sludge	84.7	95.4	97.6	100.9	93.6
40% Sludge	111.8	77.5	76.3	67.4	60.7
20%Sulfur + 5%mod.Eva	85.5	54.8	64.6	60	48.1
30%Sulfur + 5%mod.Eva	85.2	60.6	56.3	60.8	48.9

Table A 10: Viscosity (cp) at 135^{o}C (20rpm) for Heavy oil fly ash (HOFA) blends

Viscosity(cP)	0%(HOFA)	10%(HOFA)	15%(HOFA)	20%(HOFA)	25%(HOFA)
0%sulfur	571	1375	1775	2100	4500
10% sulfur	562.5	937.5	1300	2213	3412
20% sulfur	312.5	537.5	750	1362	2700
30% sulfur	262.5	475	825	1237	2975
5%mod.Eva	750	1300	2025	3400	5525
10%mod.Eva	912.5	1837	2713	3900	6600
5% SBS	7988	12325	>13000	>13000	>13000
10% SBS	>13000	>13000	>13000	>13000	>13000
20% Sludge	575	1288	2388	4137	8625
40% Sludge	525	1675	3088	5900	10000^{+}
20%Sulfur + 5%mod.Eva	362.5	750	1087	1875	3625
30%Sulfur + 5%mod.Eva	350	837.5	1362	2250	3850

Table A 11: Viscosity (cp) at 135^{o}C (20rpm) for Limestone dust (LMD) blends

Viscosity(cP)	0% (LMD)	10% (LMD)	15% (LMD)	20% (LMD)	25% (LMD)
0%sulfur	571	820	850	862.5	925
10% sulfur	562.5	450	500	512.5	562.5
20% sulfur	312.5	321	352	378	362.5
30% sulfur	262.5	287.5	312.5	337.5	387.5
5%mod.Eva	750	950	987.5	1075	1188
10%mod.Eva	912.5	1150	1300	1400	1625
5% SBS	7988	4563	5063	10100	12487
10% SBS	>13000	>13000	>13000	>13000	>13000
20% Sludge	575	773	825	887.5	1000
40% Sludge	525	675	775	825	950
20%Sulfur + 5%mod.Eva	362.5	475	512.5	575	625
30%Sulfur + 5%mod.Eva	350	500	575	637.5	675

Table A 12: Viscosity (cp) at 135oC (20rpm) for Cement-Kiln-dust (CKD) blends

Viscosity(cP)	0% (CKD)	10% (CKD)	15% (CKD)	20% (CKD)	25% (CKD)
0%sulfur	571	775	875	912.5	1075
10% sulfur	562.5	437.5	500	537.5	575
20% sulfur	312.5	312.5	325	362.5	425
30% sulfur	262.5	287.5	312.5	325	375
5%mod.Eva	750	1112	1188	1225	1337
10%mod.Eva	912.5	1475	1638	2037	2300
5% SBS	7988	7875	8488	10125	11775
10% SBS	>13000	>13000	>13000	>13000	>13000
20% Sludge	575	812.5	850	1013	1125
40% Sludge	525	950	1025	1212	1450
20%Sulfur + 5%mod.Eva	362.5	525	562.5	650	787.5
30%Sulfur + 5%mod.Eva	350	562.5	662.5	662.5	775

Table A 13: Open cup Flash point (^oC) of Heavy oil fly ash (HOFA) blends

Temp. (^oC)	0%(HOFA)	10%(HOFA)	15%(HOFA)	20%(HOFA)	25%(HOFA)
0%sulfur	338	335	X	X	X
10% sulfur	210	X	X	X	X
20% sulfur	205	X	195	X	X
30% sulfur	X	185	190	X	X
5%mod.Eva	200	208	X	X	198
10%mod.Eva	200	177	X	X	185
5% SBS	330	285	X	X	250
10% SBS	330	285	X	X	290
20% Sludge	255	245	X	X	235
40% Sludge	245	250	X	X	230
20%Sulfur + 5%mod.Eva	140	185	X	X	180
30%Sulfur + 5%mod.Eva	150	185	X	X	187

Table A 14: Open cup flash point (^{o}C) of Limestone dust (CKD) blends

Temp. (^{o}C)	0% (LMD)	10% (LMD)	15% (LMD)	20% (LMD)	25% (LMD)
0%sulfur	338	345	X	X	345
10% sulfur	210	220	X	X	220
20% sulfur	205	215	X	X	215
30% sulfur	X	210	X	X	215
5%mod.Eva	200	195	X	X	197
10%mod.Eva	200	170	X	X	180
5% SBS	330	330	X	X	320
10% SBS	330	315	X	X	315
20% Sludge	255	240	X	X	240
40% Sludge	245	235	X	X	235
20%Sulfur + 5%mod.Eva	140	155	X	X	160
30%Sulfur + 5%mod.Eva	150	170	X	X	185

Table A 15: Open cup Flash point (^oC) of Cement-Kiln-dust (CKD) blends

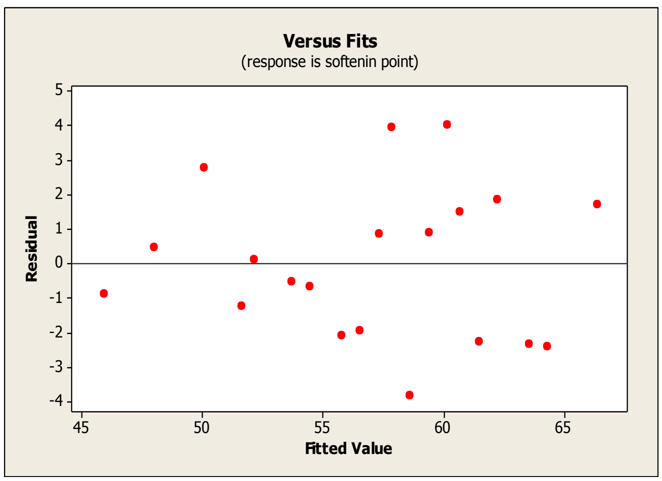
Temp. (^oC)	0% (CKD)	10% (CKD)	15% (CKD)	20% (CKD)	25% (CKD)
0%sulfur	338	342	X	X	315
10% sulfur	210	210	X	X	190
20% sulfur	205	200	X	X	170
30% sulfur	X	195	X	X	175
5%mod.Eva	200	195	X	X	205
10%mod.Eva	200	180	X	X	190
5% SBS	330	280	X	X	240
10% SBS	330	300	X	X	280
20% Sludge	255	200	X	X	200
40% Sludge	245	220	X	X	195
20%Sulfur + 5%mod.Eva	140	170	X	X	180
30%Sulfur + 5%mod.Eva	150	170	X	X	160

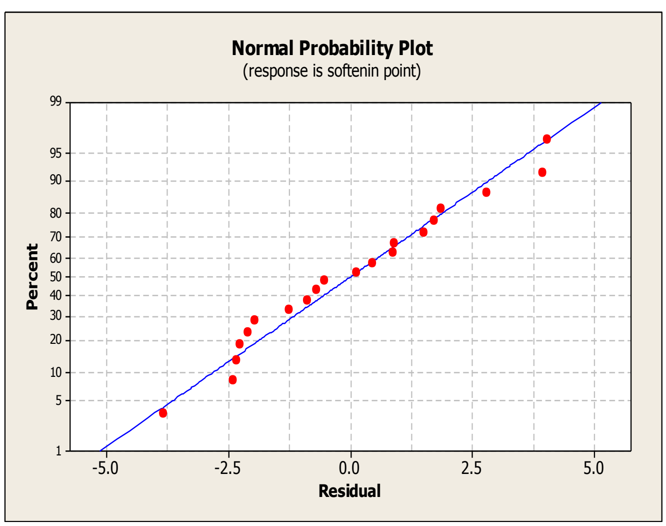
APPENDIX B

Minitab Regression model printout: SOFTENING POINT (^oC)

Sulfur-HOFA

```
softenin point  =  52.1793 - 20.7 % sulfur + 56.7905 %HOFA


Coefficients

Term          Coef  SE Coef        T      P
Constant   52.1793  1.21680  42.8825  0.000
% sulfur  -20.7000  4.66250  -4.4397  0.000
%HOFA      56.7905  6.05980   9.3717  0.000

Summary of Model

S = 2.33125       R-Sq = 86.35%        R-Sq(adj) = 84.74%
PRESS = 127.791   R-Sq(pred) = 81.12%
```

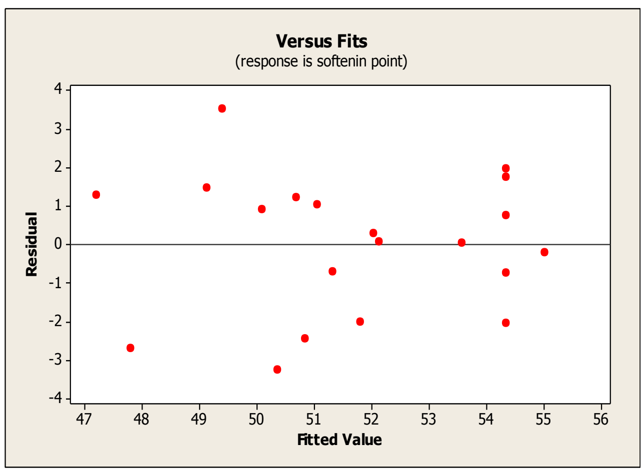


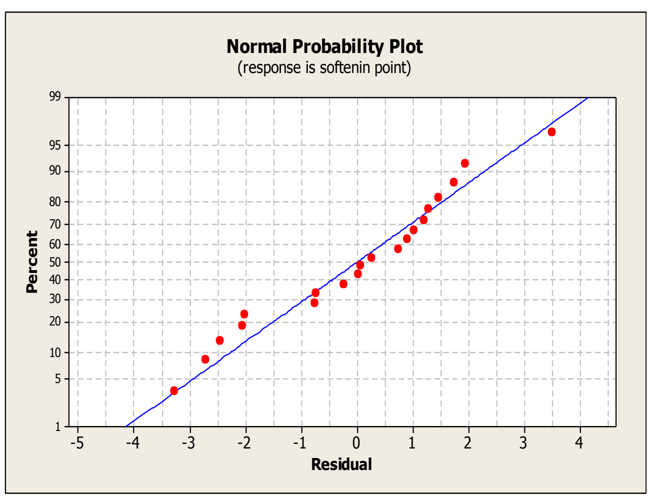


Minitab Regression model printout: SOFTENING POINT (^{o}C)

Sulfur-CKD

```
softenin point  =  54.351 - 63.3657 % sulfur + 96.2548 CKD*Sulfur + 138.5
                   %Sulfur.sq

Coefficients

Term            Coef  SE Coef        T      P
Constant      54.351   0.8451  64.3148  0.000
% sulfur     -63.366  14.0854  -4.4987  0.000
CKD*Sulfur    96.255  26.9373   3.5733  0.003
%Sulfur.sq   138.500  43.3516   3.1948  0.006

Summary of Model

S = 1.93874       R-Sq = 63.30%         R-Sq(adj) = 56.42%
PRESS = 106.634   R-Sq(pred) = 34.93%
```

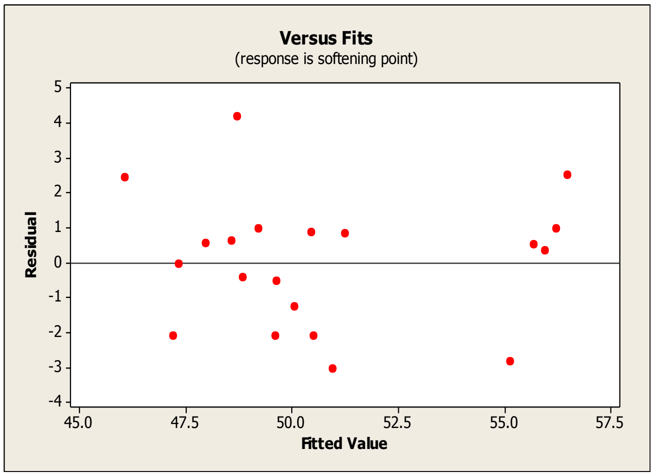


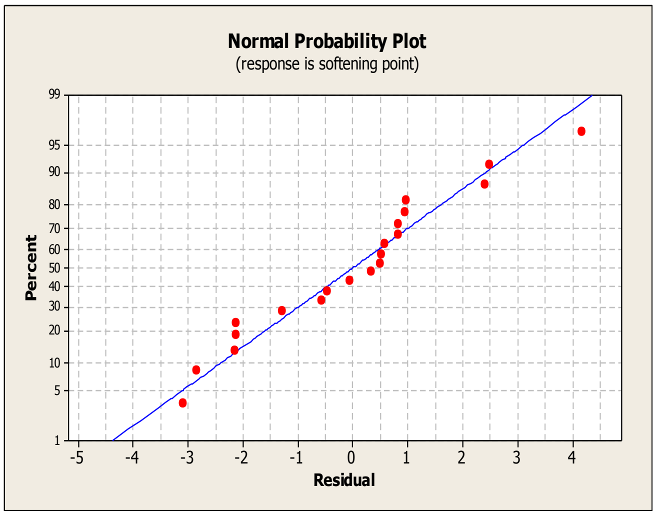


Minitab Regression model printout: SOFTENING POINT (^{o}C)

Sulfur-LMD

```
softenin point  =  55.1465 - 83.0811 % sulfur + 5.41081 %LMD + 35.8649
                   LMD*Sulfur + 189 %Sulfur.sq

Coefficients

Term             Coef  SE Coef        T      P
Constant       55.146   1.5832  34.8324  0.000
% sulfur      -83.081  16.3204  -5.0906  0.000
%LMD            5.411   9.1955   0.5884  0.565
LMD*Sulfur     35.865  49.1519   0.7297  0.477
%Sulfur.sq    189.000  47.2728   3.9981  0.001

Summary of Model

S = 2.11410       R-Sq = 75.37%        R-Sq(adj) = 68.80%
PRESS = 165.407   R-Sq(pred) = 39.22%
```

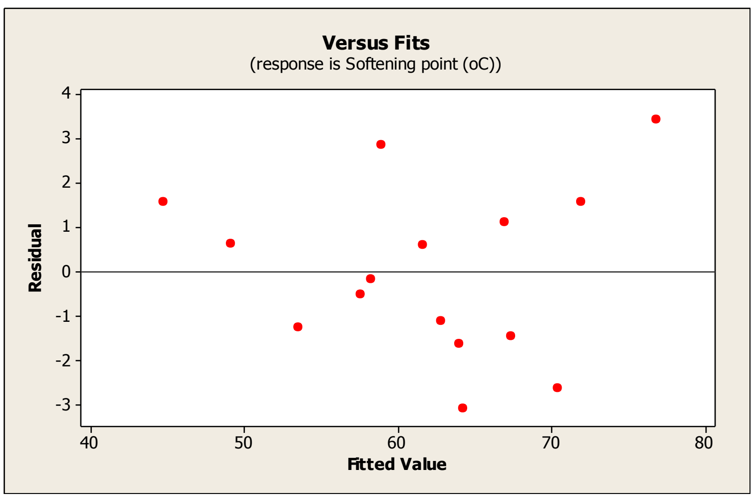


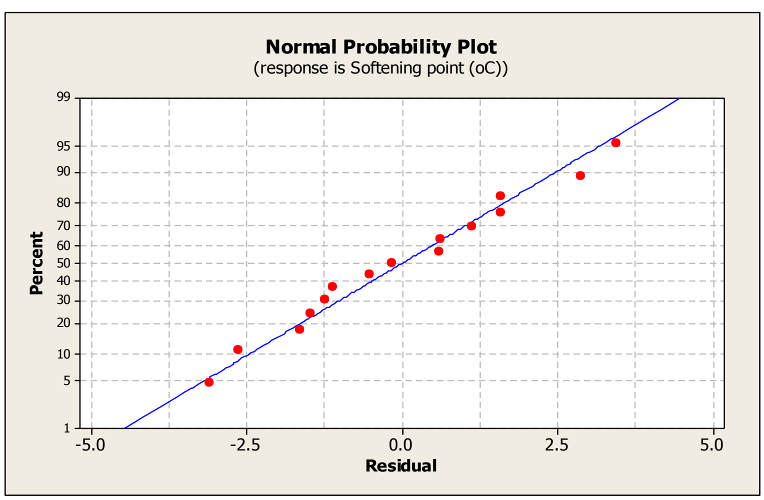


Minitab Regression model printout: SOFTENING POINT (^oC)

Sludge-HOFA

```
Softening point (oC)  =  53.5432 - 21.8514 % Sludge + 53.7387 % HOFA + 186.081
                         Sludge*HOFA


Coefficients

Term            Coef  SE Coef        T      P
Constant      53.543   1.6909  31.6655  0.000
% Sludge     -21.851   6.5488  -3.3367  0.007
% HOFA        53.739  10.2905   5.2222  0.000
Sludge*HOFA  186.081  39.8549   4.6690  0.001


Summary of Model

S = 2.16834      R-Sq = 95.21%        R-Sq(adj) = 93.90%
PRESS = 124.154  R-Sq(pred) = 88.50%
```

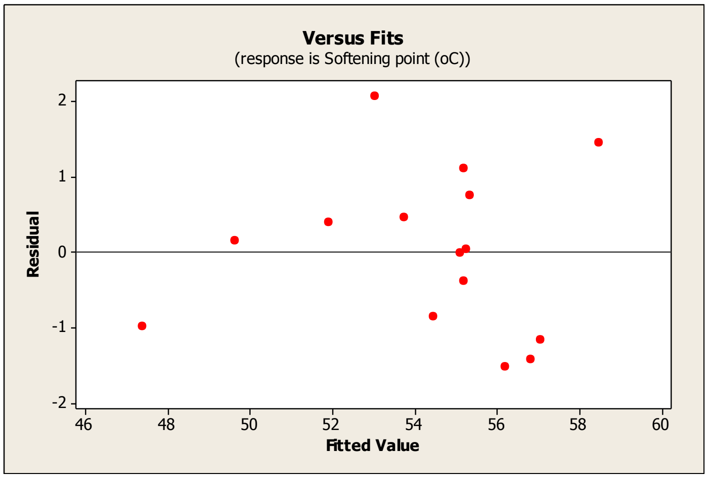


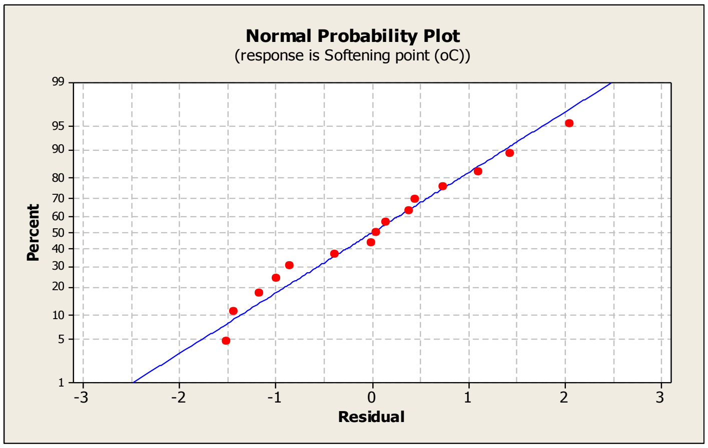


Minitab Regression model printout: SOFTENING POINT (^oC)

Sludge-CKD

```
Softening point (oC)  =  51.9125 - 11.2973 % Sludge + 33.5733 % CKD + 77.8378
                         Sludge*CKD - 81.7084 CKD.sq


Coefficients

Term            Coef  SE Coef        T      P
Constant     51.9125   1.0487  49.5024  0.000
% Sludge    -11.2973   3.8174  -2.9594  0.014
% CKD        33.5733  13.2818   2.5278  0.030
Sludge*CKD   77.8378  23.2320   3.3505  0.007
CKD.sq      -81.7084  48.1817  -1.6958  0.121


Summary of Model

S = 1.26396      R-Sq = 87.92%        R-Sq(adj) = 83.08%
PRESS = 47.5836  R-Sq(pred) = 64.01%
```

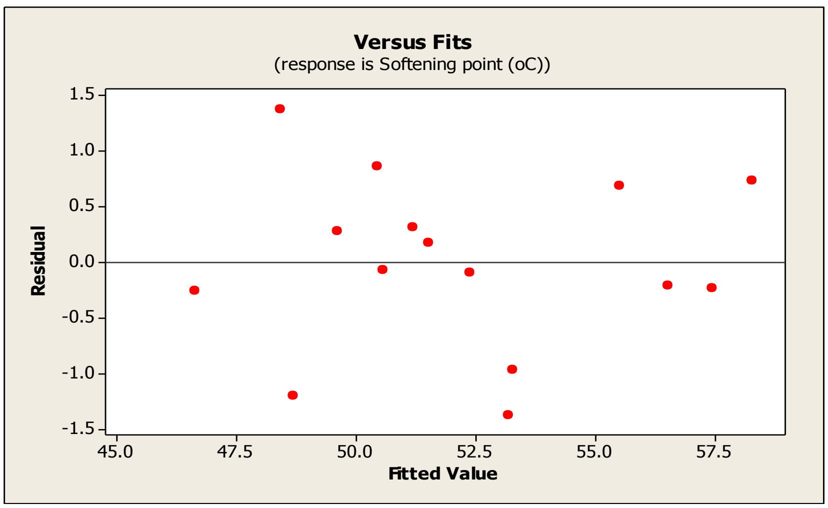


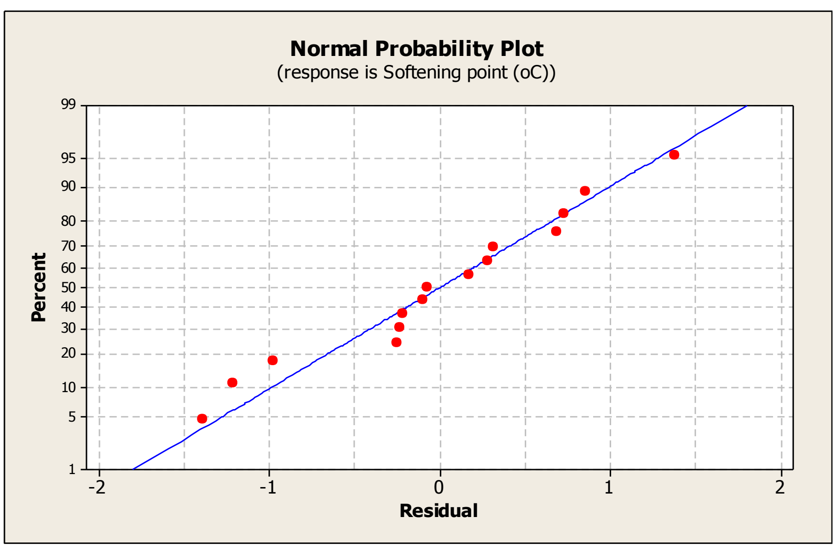


Minitab Regression model printout: SOFTENING POINT (^oC)

Sludge-LMD

```
Softening point (oC)  =  53.0103 - 17.2 % Sludge + 19.1171 % LMD


Coefficients

Term           Coef  SE Coef        T      P
Constant    53.0103  0.68730  77.1281  0.000
% Sludge   -17.2000  1.85488  -9.2728  0.000
% LMD       19.1171  3.52115   5.4292  0.000


Summary of Model

S = 1.17313      R-Sq = 90.59%        R-Sq(adj) = 89.02%
PRESS = 25.0358  R-Sq(pred) = 85.73%
```

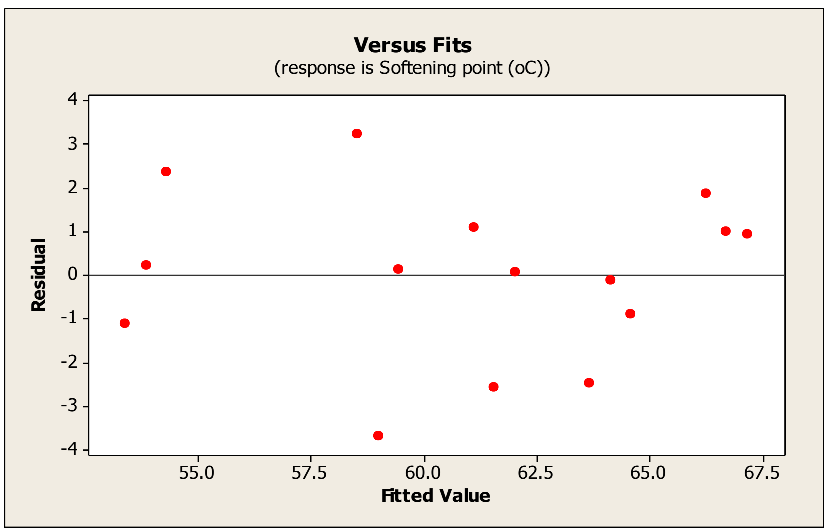


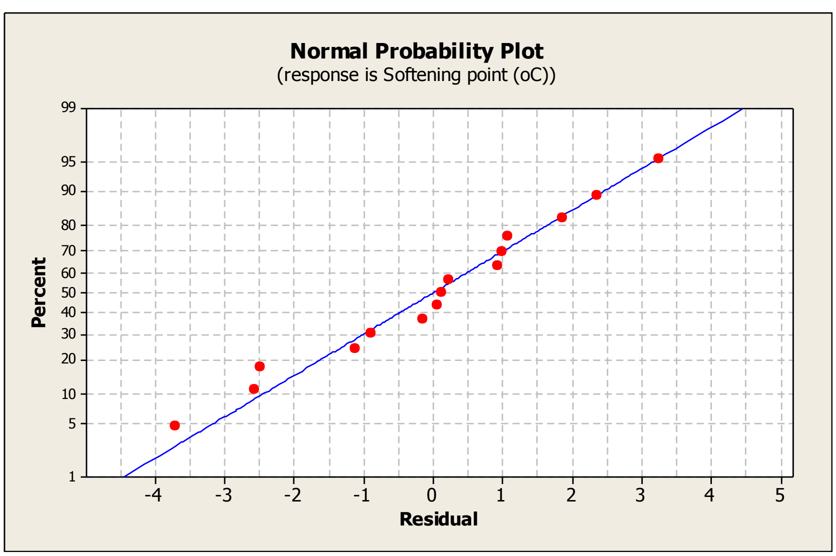


Minitab Regression model printout: SOFTENING POINT (^oC)

Mod.Eva - HOFA

```
Softening point (oC)  =  53.4184 + 9.2 % M.Eva + 51.2973 % HOFA


Coefficients

Term          Coef  SE Coef        T      P
Constant   53.4184   1.2115  44.0926  0.000
% M.Eva     9.2000  13.0784   0.7035  0.495
% HOFA     51.2973   6.2067   8.2648  0.000


Summary of Model

S = 2.06787      R-Sq = 85.15%         R-Sq(adj) = 82.67%
PRESS = 77.8851  R-Sq(pred) = 77.46%
```

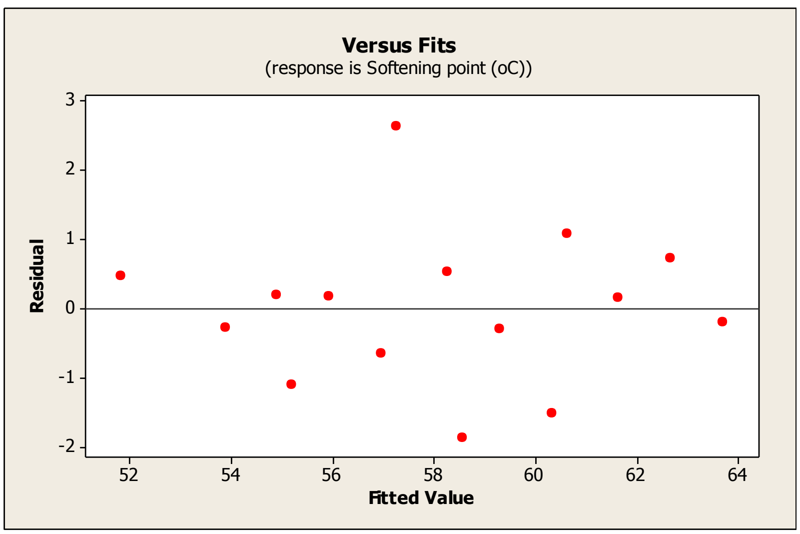


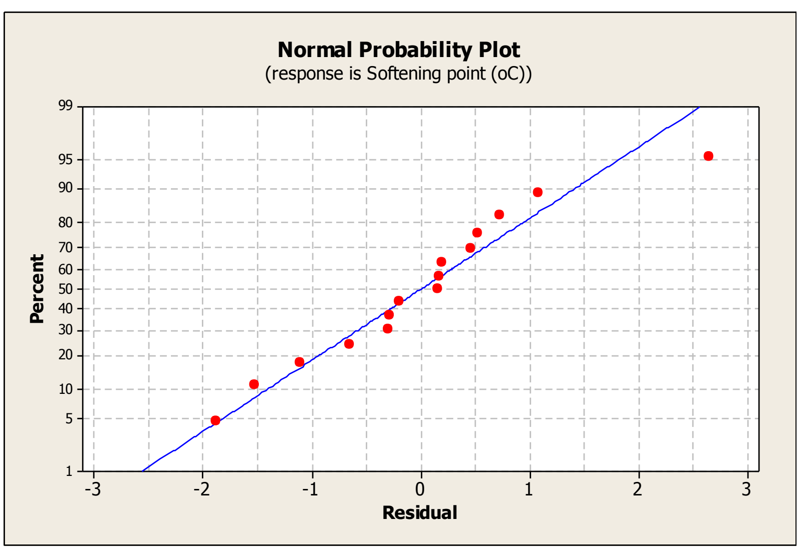


Minitab Regression model printout: SOFTENING POINT (^{o}C)

Mod.Eva - CKD

```
Softening point (oC)  =  51.839 + 67.4 % M.Eva + 20.4595 % CKD


Coefficients

Term          Coef  SE Coef        T      P
Constant   51.8390  0.69685  74.3904  0.000
% M.Eva    67.4000  7.52261   8.9597  0.000
% CKD      20.4595  3.57007   5.7308  0.000


Summary of Model

S = 1.18943      R-Sq = 90.41%        R-Sq(adj) = 88.81%
PRESS = 26.6447  R-Sq(pred) = 84.95%
```

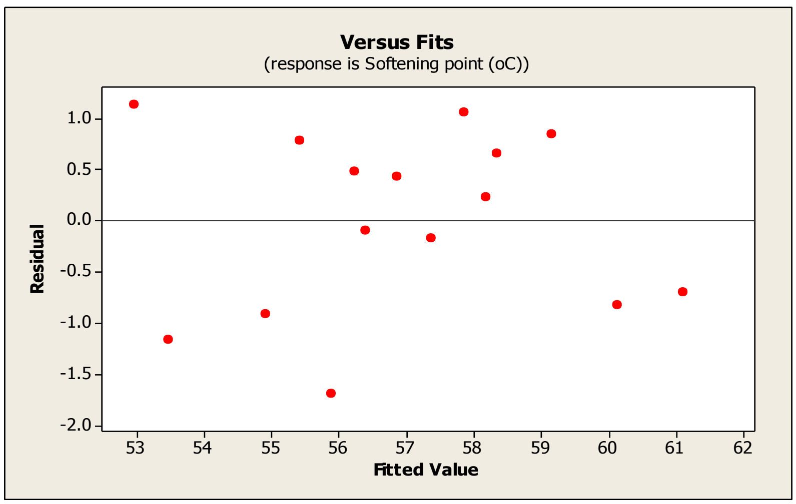


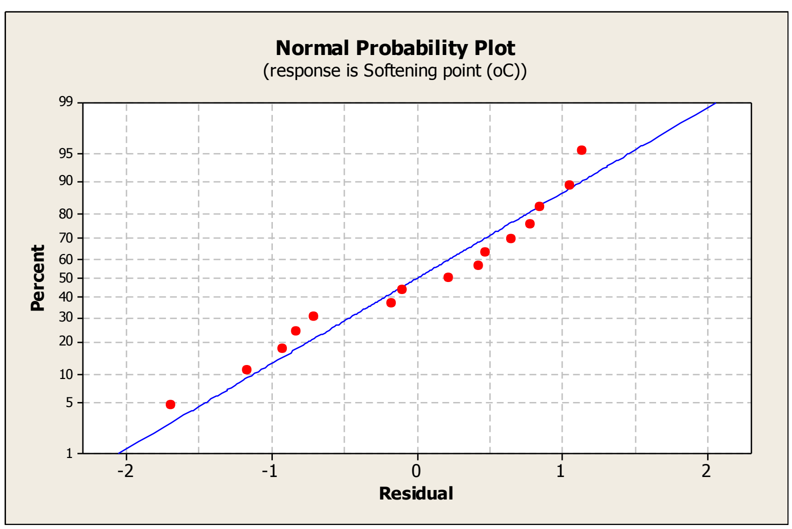


Minitab Regression model printout: SOFTENING POINT (^{o}C)

Mod.Eva - LMD

```
Softening point (oC)  =  53.4656 - 47.6 % M.Eva + 19.5315 % LMD + 752 M.Eva.sq


Coefficients

Term          Coef  SE Coef        T      P
Constant    53.466    0.611  87.4941  0.000
% M.Eva    -47.600   22.708  -2.0962  0.060
% LMD       19.532    2.989   6.5347  0.000
M.Eva.sq   752.000  218.167   3.4469  0.005


Summary of Model

S = 0.995793      R-Sq = 87.03%         R-Sq(adj) = 83.49%
PRESS = 21.2463  R-Sq(pred) = 74.73%
```

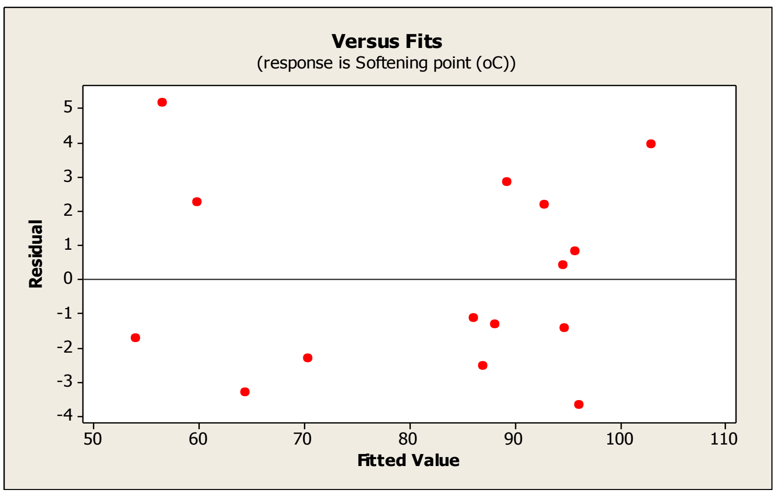


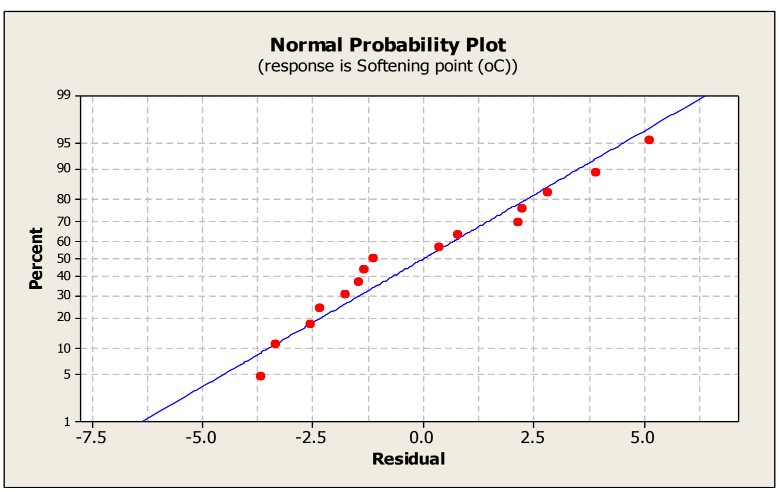


Minitab Regression model printout: SOFTENING POINT (^oC)

SBS - HOFA

```
Softening point (oC)  =  54.0416 + 877.718 % SBS - 942.271 SBS*HOFA - 3884
                         SBS.sq + 262.161 HOFA.sq


Coefficients

Term           Coef  SE Coef        T      P
Constant      54.04    2.090  25.8605  0.000
% SBS        877.72   80.211  10.9427  0.000
SBS*HOFA    -942.27  223.139  -4.2228  0.002
SBS.sq     -3884.00  709.789  -5.4720  0.000
HOFA.sq      262.16   55.775   4.7003  0.001

Summary of Model

S = 3.23973       R-Sq = 97.28%        R-Sq(adj) = 96.19%
PRESS = 344.269   R-Sq(pred) = 91.08%
```

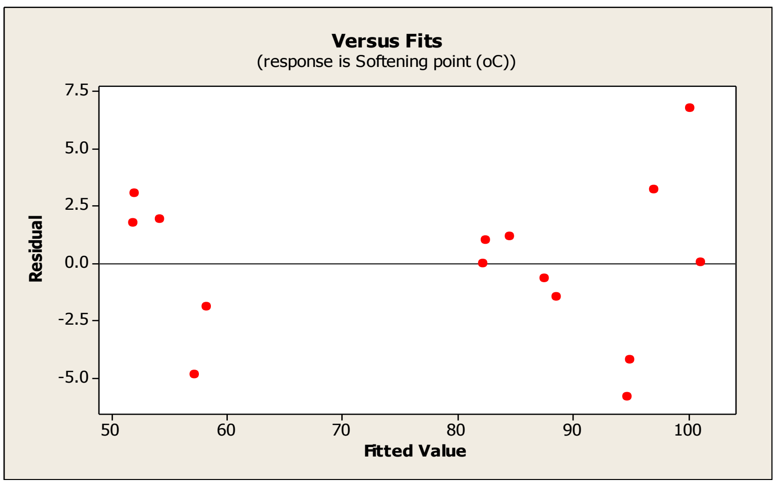


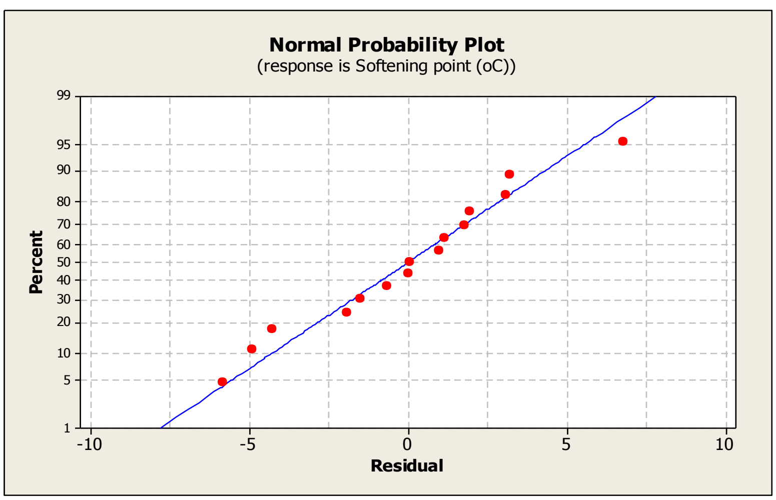


Minitab Regression model printout: SOFTENING POINT (^oC)

SBS - CKD

```
Softening point (oC)  =  57.189 + 786 % SBS - 92.2543 % CKD - 3568 SBS.sq +
                         385.43 CKD.sq


Coefficients

Term            Coef  SE Coef        T      P
Constant       57.19    2.684  21.3111  0.000
% SBS         786.00   90.536   8.6816  0.000
% CKD         -92.25   39.084  -2.3604  0.040
SBS.sq      -3568.00  869.844  -4.1019  0.002
CKD.sq        385.43  151.346   2.5467  0.029

Summary of Model

S = 3.97028      R-Sq = 96.93%        R-Sq(adj) = 95.70%
PRESS = 409.955  R-Sq(pred) = 92.01%
```

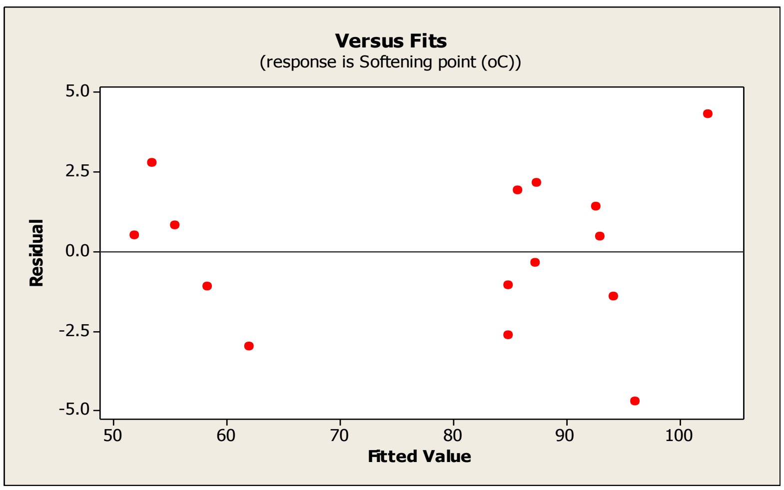


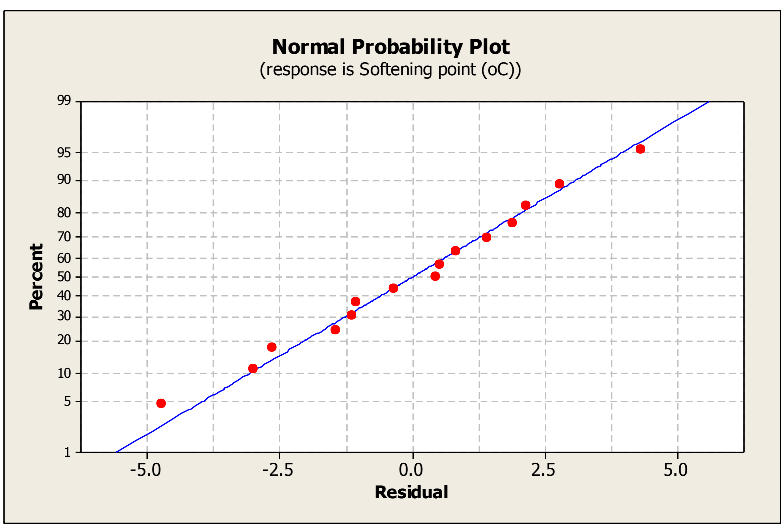


Minitab Regression model printout: SOFTENING POINT (^oC)

SBS - LMD

```
Softening point (oC)  =  51.7853 + 910.423 % SBS - 808.732 SBS*LMD - 4024
                         SBS.sq + 163.508 LMD.sq


Coefficients

Term           Coef  SE Coef        T      P
Constant      51.79    1.832  28.2670  0.000
% SBS        910.42   70.318  12.9472  0.000
SBS*LMD     -808.73  195.619  -4.1342  0.002
SBS.sq     -4024.00  622.249  -6.4669  0.000
LMD.sq       163.51   48.896   3.3440  0.007

Summary of Model

S = 2.84016       R-Sq = 98.19%        R-Sq(adj) = 97.46%
PRESS = 337.665  R-Sq(pred) = 92.42%
```

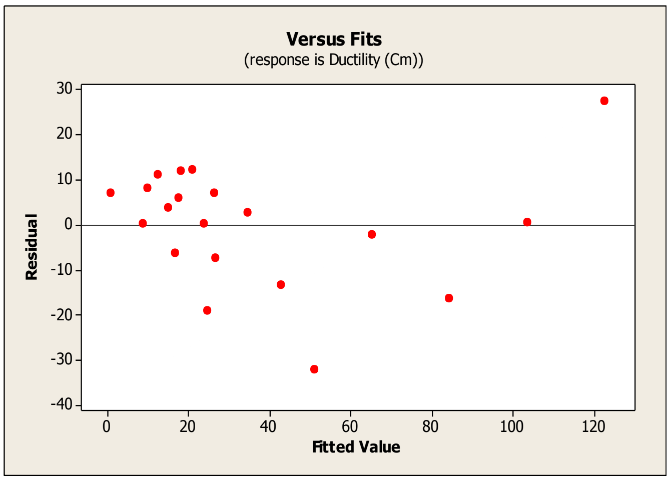


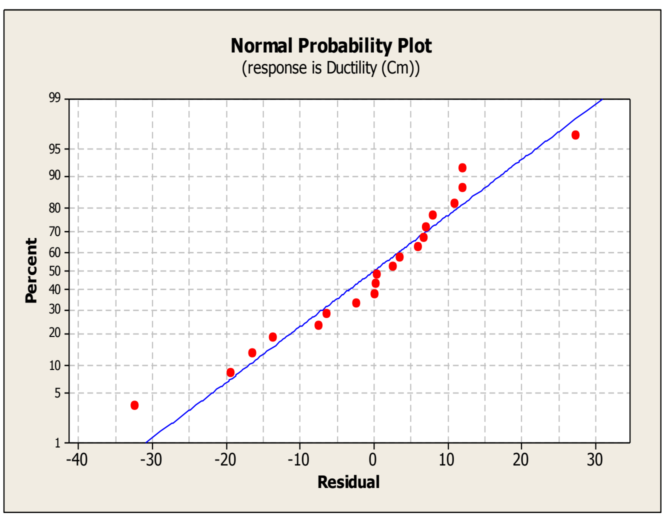


Minitab Regression model printout: DUCTILITY (cm)

Sulfur-HOFA

```
Ductility (Cm)  =  122.644 - 191.178 % sulfur - 866.764 %HOFA + 1083.7
                   HOFA*Sulfur + 1518.02 %HOFA.sq

Coefficients

Term            Coef  SE Coef        T      P
Constant      122.64   11.281  10.8722  0.000
% sulfur     -191.18   57.030  -3.3522  0.004
%HOFA        -866.76  137.504  -6.3036  0.000
HOFA*Sulfur  1083.70  347.074   3.1224  0.007
%HOFA.sq     1518.02  492.821   3.0803  0.008


Summary of Model

S = 14.9282      R-Sq = 86.20%        R-Sq(adj) = 82.52%
PRESS = 8938.30  R-Sq(pred) = 63.10%
```

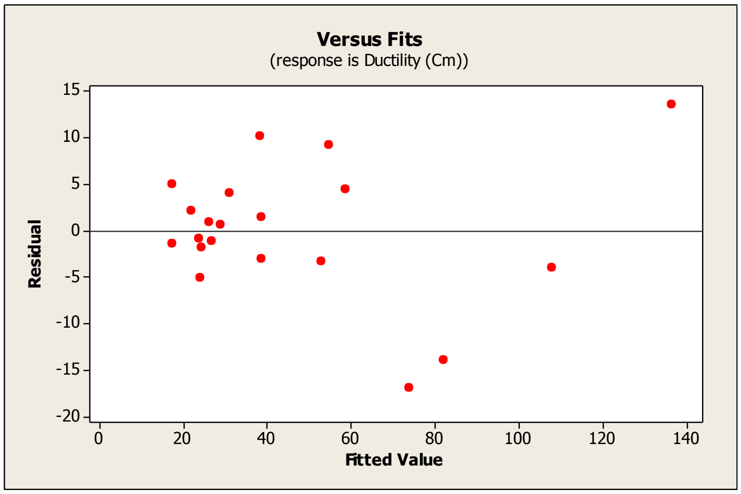


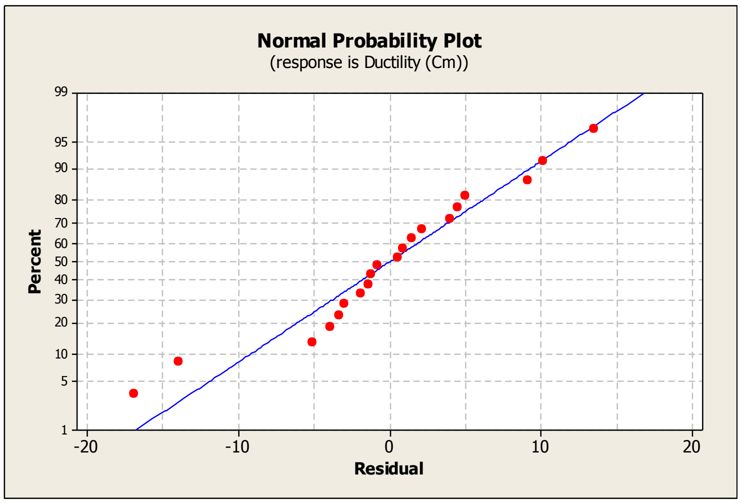


Minitab Regression model printout: DUCTILITY (cm)

Sulfur-CKD

```
Ductility (Cm)  =  135.225 - 259.959 % Sulfur - 761.723 % CKD + 947.568
                   Sulfur*CKD + 1358.76 CKD.Sq


Coefficients

Term            Coef  SE Coef         T      P
Constant      135.22    6.235   21.6886  0.000
% Sulfur     -259.96   31.521   -8.2472  0.000
% CKD        -761.72   75.999  -10.0228  0.000
Sulfur*CKD    947.57  191.829    4.9396  0.000
CKD.Sq       1358.76  272.384    4.9884  0.000

Summary of Model

S = 8.25089       R-Sq = 95.01%        R-Sq(adj) = 93.69%
PRESS = 2719.72   R-Sq(pred) = 86.72%
```

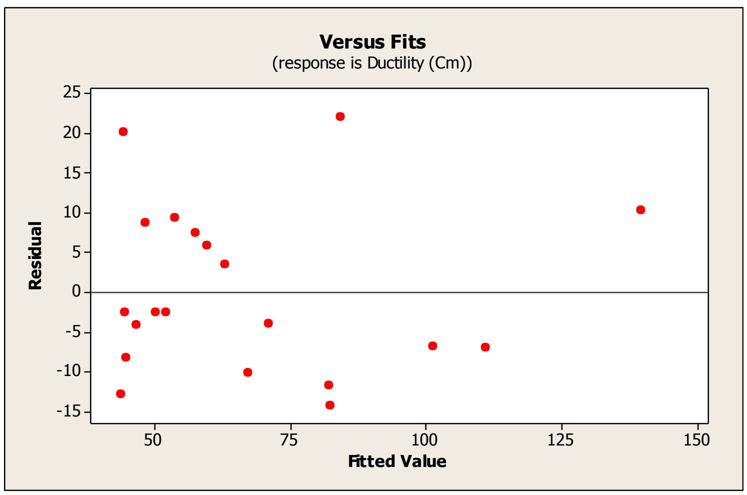


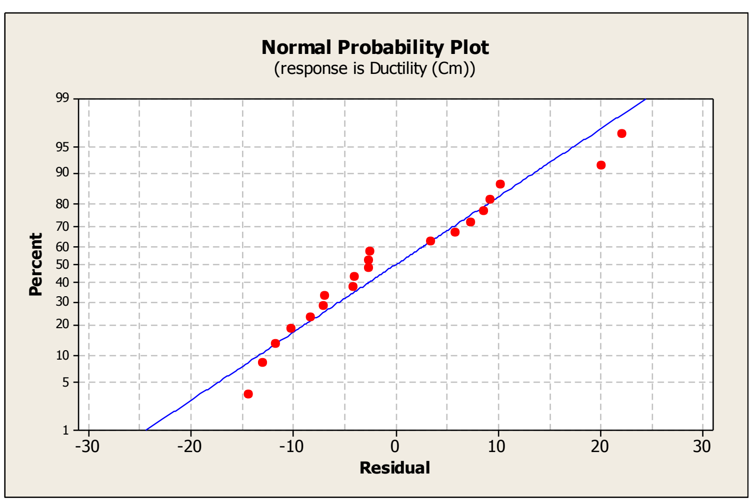


Minitab Regression model printout: DUCTILITY (cm)

Sulfur-LMD

```
Ductility (Cm)  =  139.721 - 286.819 % sulfur - 383.249 %LMD + 1159.14
                   LMD*Sulfur

Coefficients

Term            Coef  SE Coef        T      P
Constant      139.72    8.175  17.0914  0.000
% sulfur     -286.82   43.697  -6.5639  0.000
%LMD         -383.25   49.751  -7.7034  0.000
LMD*Sulfur   1159.14  265.929   4.3588  0.000

Summary of Model

S = 11.4381       R-Sq = 85.99%         R-Sq(adj) = 83.37%

PRESS = 3627.12   R-Sq(pred) = 75.73%
```

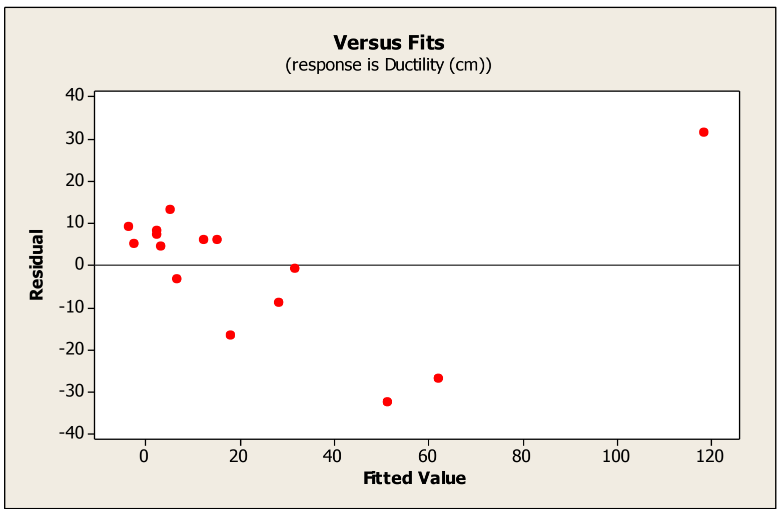


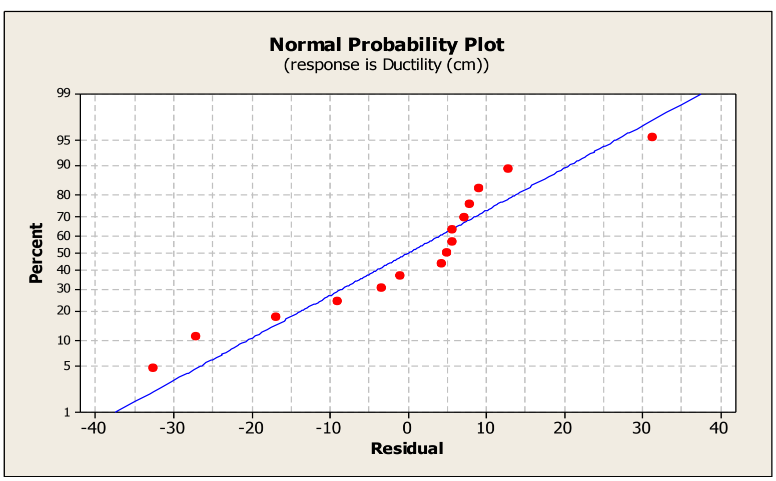


Minitab Regression model printout: DUCTILITY (cm)

Sludge-HOFA

```
Ductility (cm)  =  118.57 - 348.484 % Sludge - 811.198 % HOFA + 1014.53
                   Sludge*HOFA + 329.25 Sludge.sq + 1400.64 HOFA.sq

Coefficients

Term            Coef  SE Coef         T      P
Constant      118.57   17.058   6.95097  0.000
% Sludge     -348.48  125.591  -2.77476  0.022
% HOFA       -811.20  210.996  -3.84461  0.004
Sludge*HOFA  1014.53  369.065   2.74891  0.023
Sludge.sq     329.25  274.947   1.19750  0.262
HOFA.sq      1400.64  765.418   1.82990  0.101

Summary of Model

S = 20.0793      R-Sq = 80.45%        R-Sq(adj) = 69.59%
PRESS = 20233.1  R-Sq(pred) = -8.99%
```

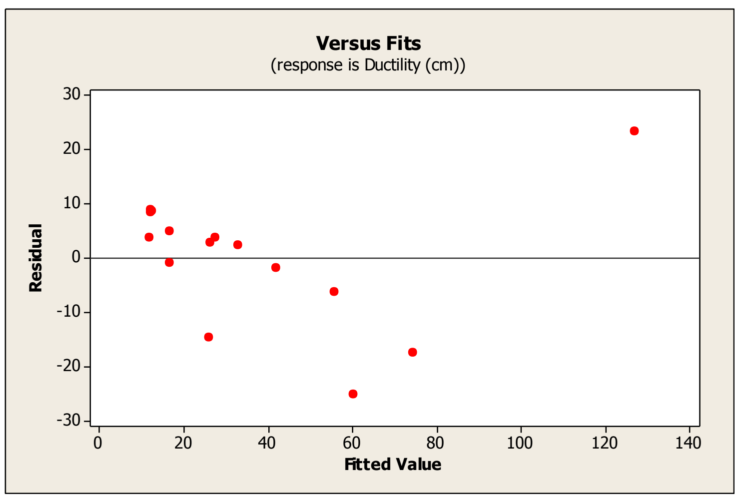


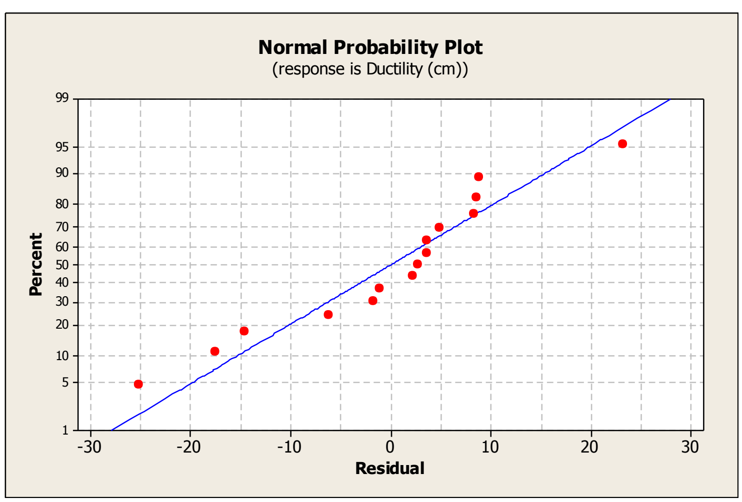


Minitab Regression model printout: DUCTILITY (cm)

Sludge-CKD

```
Ductility (cm)  =  126.822 - 418.681 % Sludge - 621.452 % CKD + 927.365
                   Sludge*CKD + 425.25 Sludge.sq + 980.795 CKD.sq

Coefficients

Term             Coef  SE Coef         T      P
Constant      126.822   12.703   9.98392  0.000
% Sludge     -418.681   93.523  -4.47676  0.002
% CKD        -621.452  157.122  -3.95523  0.003
Sludge*CKD    927.365  274.830   3.37432  0.008
Sludge.sq     425.250  204.744   2.07698  0.068
CKD.sq        980.795  569.982   1.72075  0.119

Summary of Model

S = 14.9524      R-Sq = 87.45%          R-Sq(adj) = 80.48%
PRESS = 11753.2  R-Sq(pred) = 26.70%
```

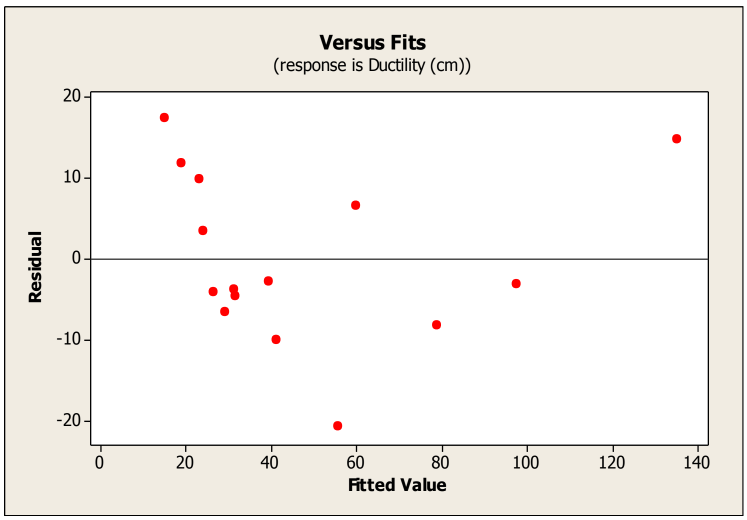


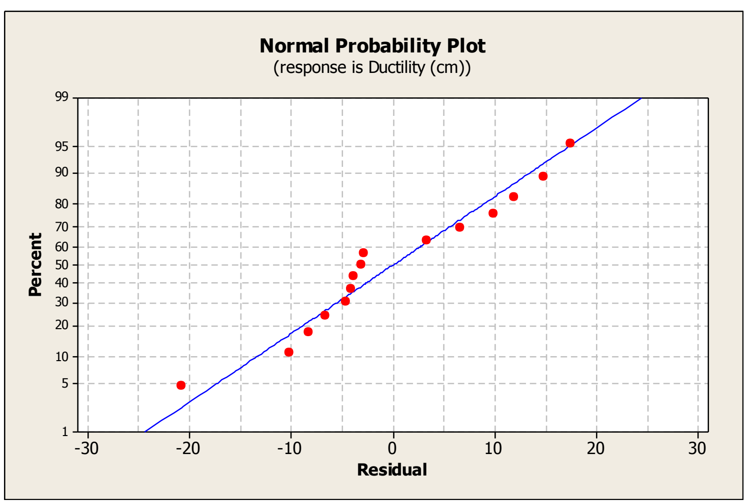


Minitab Regression model printout: DUCTILITY (cm)

Sludge-LMD

```
Ductility (cm)  =  135.188 - 504.07 % Sludge - 376.342 % LMD + 1066.22
                   Sludge*LMD + 534.5 Sludge.sq

Coefficients

Term           Coef  SE Coef        T      P
Constant     135.19    9.922  13.6244  0.000
% Sludge    -504.07   77.491  -6.5049  0.000
% LMD       -376.34   58.796  -6.4008  0.000
Sludge*LMD  1066.22  227.717   4.6822  0.001
Sludge.sq    534.50  169.645   3.1507  0.010

Summary of Model

S = 12.3891       R-Sq = 91.14%        R-Sq(adj) = 87.60%
PRESS = 5544.26   R-Sq(pred) = 68.01%
```

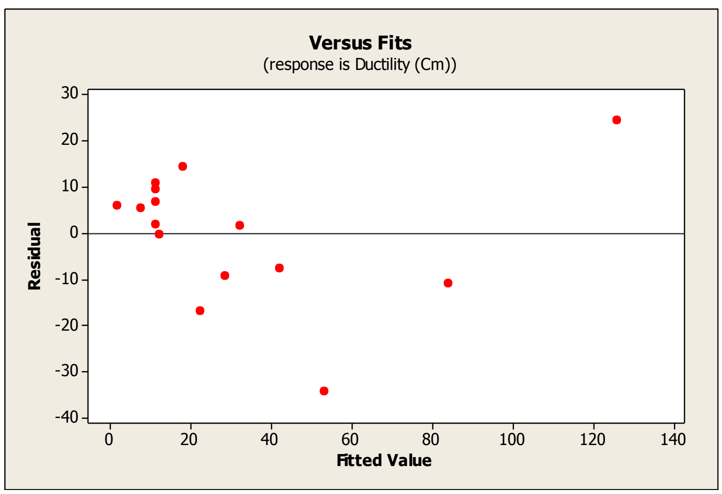


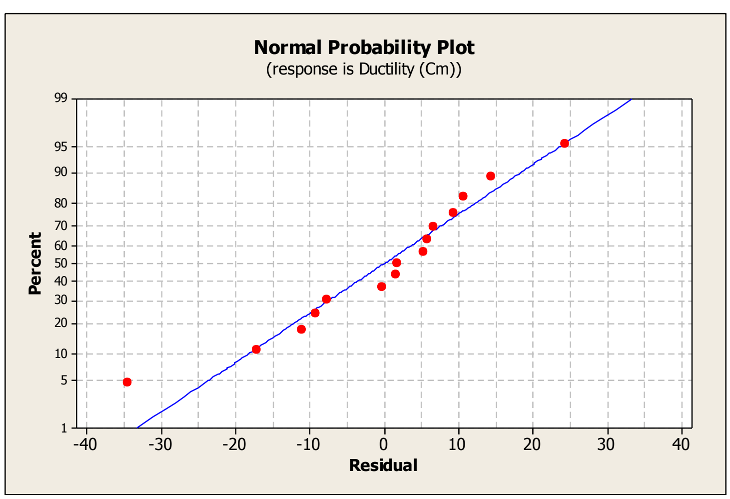


Minitab Regression model printout: DUCTILITY (cm)

Mod.Eva - HOFA

```
Ductility (Cm)  =  125.729 + 1520.17 HOFA.sq - 835.486 % M.Eva - 875.356 % HOFA
                   + 4169.19 M.Eva*HOFA


Coefficients

Term            Coef  SE Coef         T      P
Constant      125.73    14.02   8.96996  0.000
HOFA.sq      1520.17   643.99   2.36054  0.040
% M.Eva      -835.49   204.09  -4.09368  0.002
% HOFA       -875.36   177.52  -4.93094  0.001
M.Eva*HOFA   4169.19  1242.06   3.35666  0.007

Summary of Model

S = 16.8939      R-Sq = 84.80%        R-Sq(adj) = 78.72%
PRESS = 11659.3  R-Sq(pred) = 37.90%
```

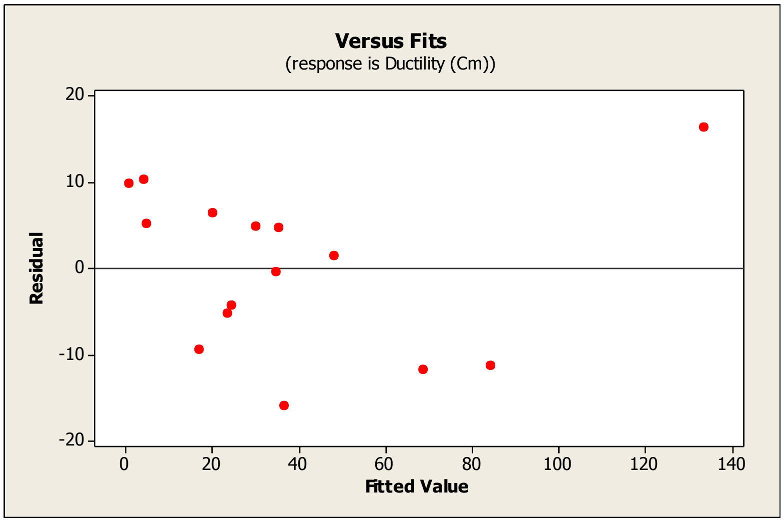


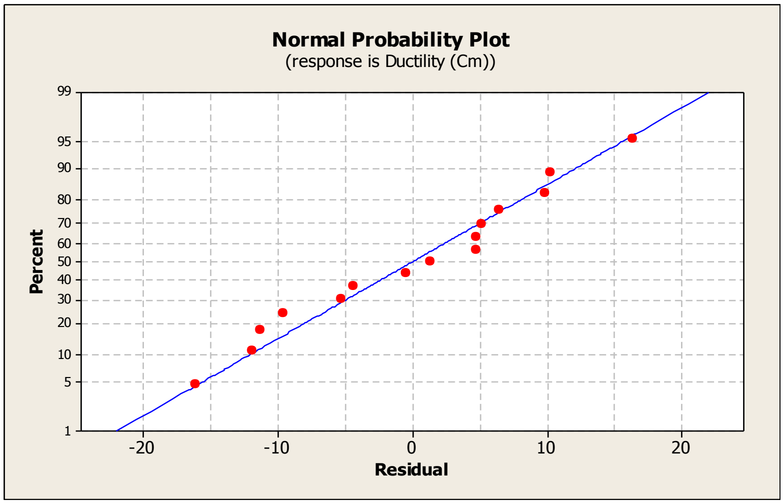


Minitab Regression model printout: DUCTILITY (cm)

Mod.Eva - CKD

```
Ductility (Cm)  =  133.677 - 987.622 % M.Eva - 804.793 % CKD + 3417.3 M.Eva*CKD
                   + 1564.21 CKD.sq


Coefficients

Term           Coef  SE Coef        T      P
Constant     133.68    9.261  14.4349  0.000
% M.Eva     -987.62  134.842  -7.3243  0.000
% CKD       -804.79  117.288  -6.8617  0.000
M.Eva*CKD   3417.30  820.621   4.1643  0.002
CKD.sq      1564.21  425.480   3.6764  0.004

Summary of Model

S = 11.1616      R-Sq = 93.25%        R-Sq(adj) = 90.55%
PRESS = 4784.71  R-Sq(pred) = 74.09%
```

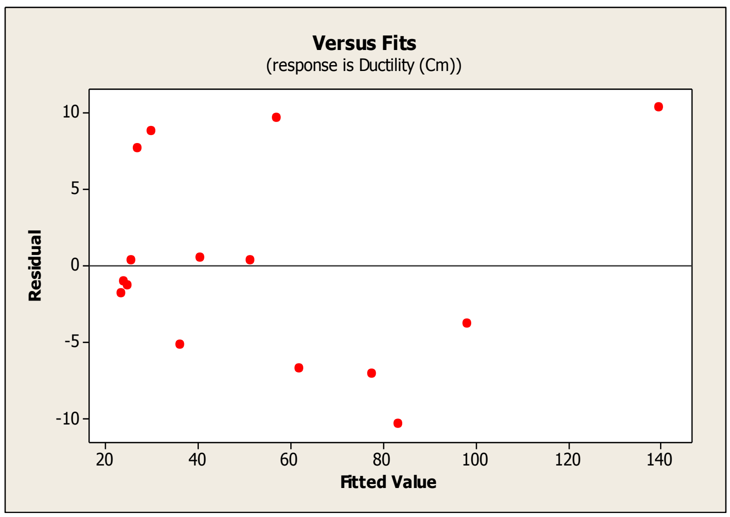


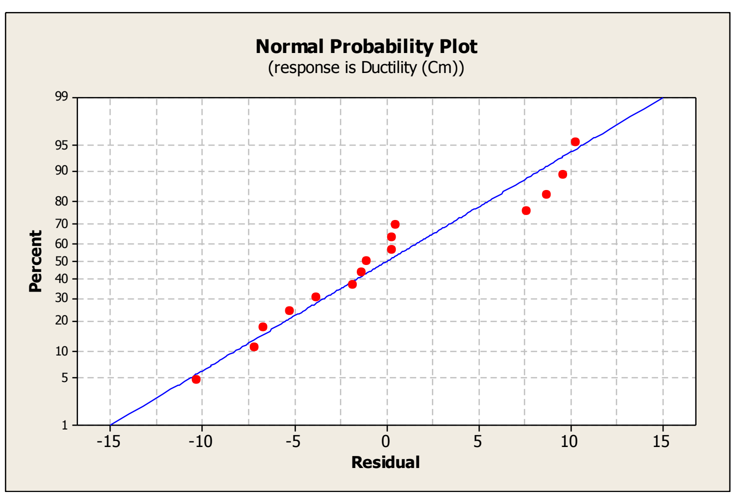


Minitab Regression model printout: DUCTILITY (cm)

Mod.Eva - LMD

```
Ductility (Cm)  =  139.729 - 1128.3 % M.Eva - 414.09 % LMD + 3997.84 M.Eva*LMD


Coefficients

Term            Coef  SE Coef         T      P
Constant      139.73    5.671   24.6388  0.000
% M.Eva     -1128.30   87.856  -12.8425  0.000
% LMD        -414.09   34.513  -11.9980  0.000
M.Eva*LMD    3997.84  534.676    7.4771  0.000


Summary of Model

S = 7.27238       R-Sq = 96.52%        R-Sq(adj) = 95.57%
PRESS = 1774.83   R-Sq(pred) = 89.38%
```

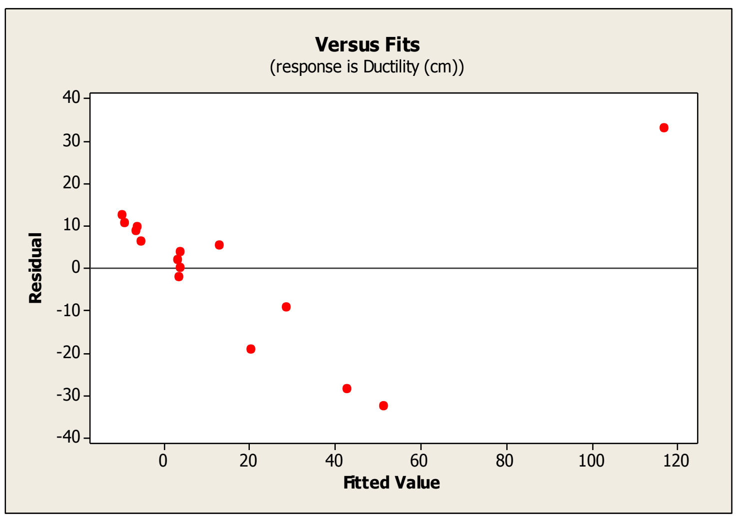


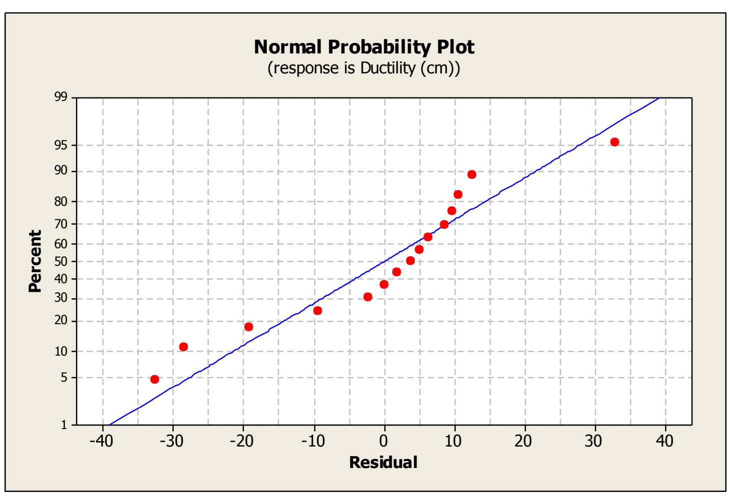


Minitab Regression model printout: DUCTILITY (cm)

SBS - HOFA

```
Ductility (cm)  =  117.04 - 1829.78 % SBS - 791.25 % HOFA + 5192.7 SBS*HOFA +
                   6984 SBS.sq + 1353.89 HOFA.sq

Coefficients

Term           Coef  SE Coef         T      P
Constant     117.04    17.80   6.57709  0.000
% SBS      -1829.78   524.07  -3.49150  0.007
% HOFA      -791.25   220.11  -3.59476  0.006
SBS*HOFA    5192.70  1540.04   3.37179  0.008
SBS.sq      6984.00  4589.22   1.52183  0.162
HOFA.sq     1353.89   798.49   1.69557  0.124

Summary of Model

S = 20.9468      R-Sq = 79.94%          R-Sq(adj) = 68.79%
PRESS = 22353.4  R-Sq(pred) = -13.57%
```

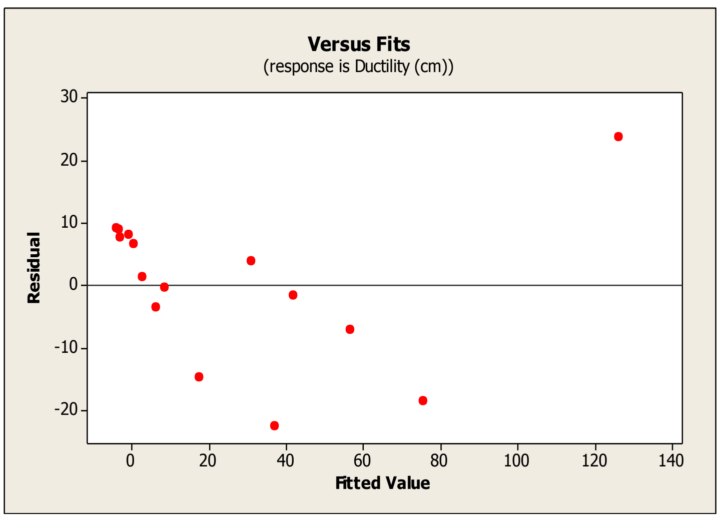


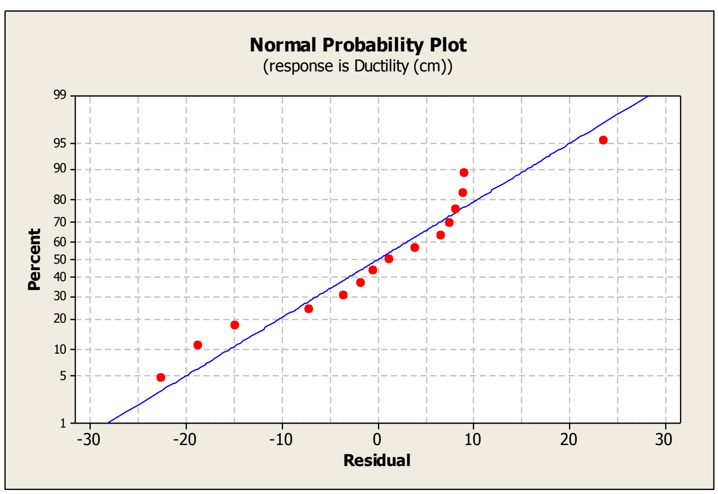


Minitab Regression model printout: DUCTILITY (cm)

SBS - CKD

```
Ductility (cm)  =  126.352 - 2333.04 % SBS - 590.505 % CKD + 4400.27 SBS*CKD +
                   10972 SBS.sq + 837.712 CKD.sq


Coefficients

Term          Coef  SE Coef         T      P
Constant     126.4    12.84   9.84014  0.000
% SBS      -2333.0   378.15  -6.16955  0.000
% CKD       -590.5   158.83  -3.71790  0.005
SBS*CKD     4400.3  1111.26   3.95973  0.003
SBS.sq     10972.0  3311.47   3.31333  0.009
CKD.sq       837.7   576.17   1.45393  0.180

Summary of Model

S = 15.1147       R-Sq = 90.32%        R-Sq(adj) = 84.94%
PRESS = 11844.8   R-Sq(pred) = 44.22%
```

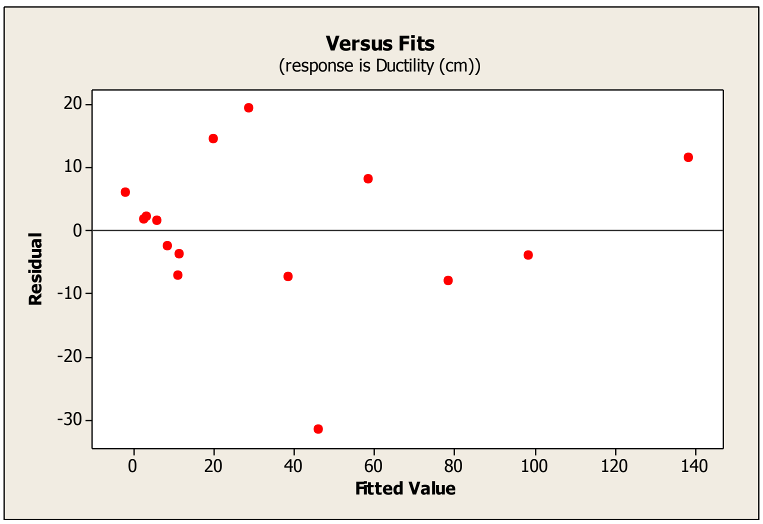


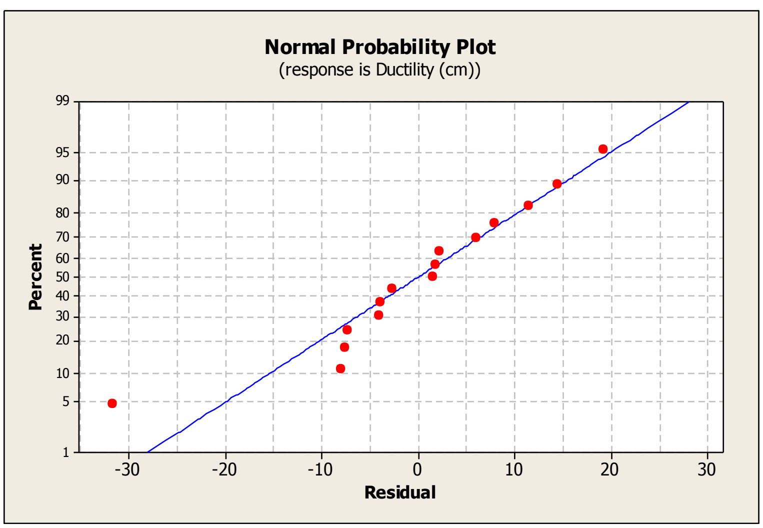


Minitab Regression model printout: DUCTILITY (cm)

SBS - LMD

```
Ductility (cm)  =  138.494 - 2291.54 % SBS - 399.955 % LMD + 4532.43 SBS*LMD +
                   8860 SBS.sq

Coefficients

Term            Coef  SE Coef        T      P
Constant      138.49    11.41  12.1349  0.000
% SBS       -2291.54   356.52  -6.4275  0.000
% LMD        -399.95    67.63  -5.9141  0.000
SBS*LMD      4532.43  1047.68   4.3262  0.001
SBS.sq       8860.00  3122.02   2.8379  0.018

Summary of Model

S = 14.2500       R-Sq = 92.09%        R-Sq(adj) = 88.92%
PRESS = 5508.51   R-Sq(pred) = 78.53%
```

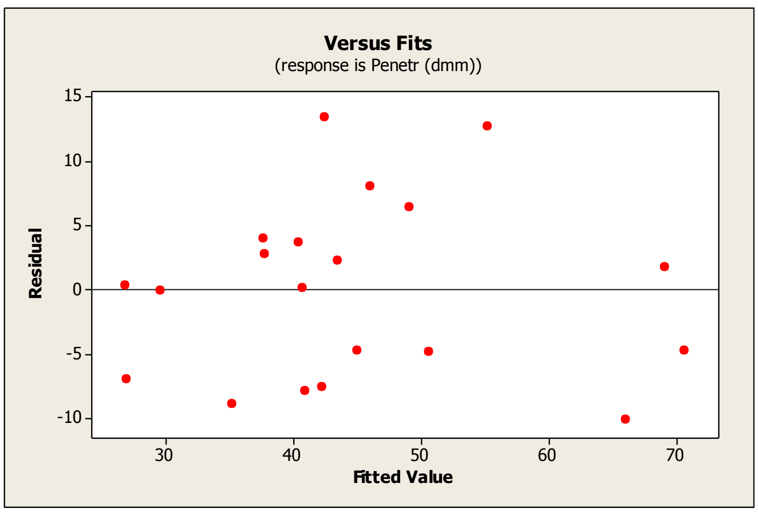


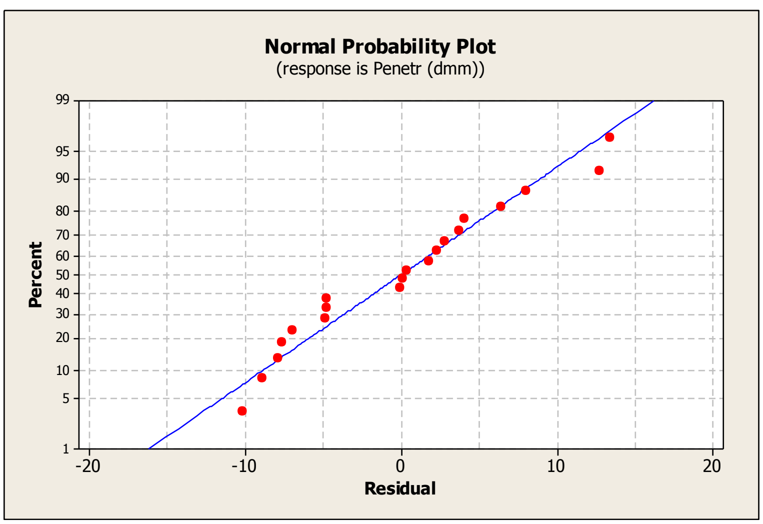


Minitab Regression model printout: PENETRATION (dmm)

Sulfur-HOFA

```
Penetr (dmm)  =  55.1806 + 139.27 % Sulfur - 257.814 % HOFA + 580.015 HOFA.Sq -
                309.5 Sulfur.Sq


Coefficients

Term            Coef  SE Coef        T      P
Constant      55.181    4.832  11.4193  0.000
% Sulfur     139.270   54.683   2.5469  0.022
% HOFA      -257.814   66.598  -3.8712  0.002
HOFA.Sq      580.015  257.890   2.2491  0.040
Sulfur.Sq   -309.500  174.678  -1.7718  0.097

Summary of Model

S = 7.81185       R-Sq = 76.63%        R-Sq(adj) = 70.40%
PRESS = 1817.10   R-Sq(pred) = 53.61%
```

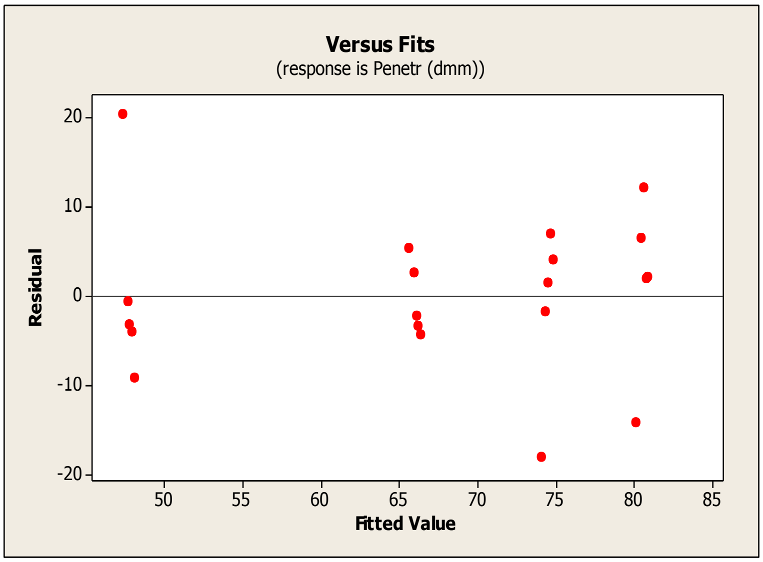


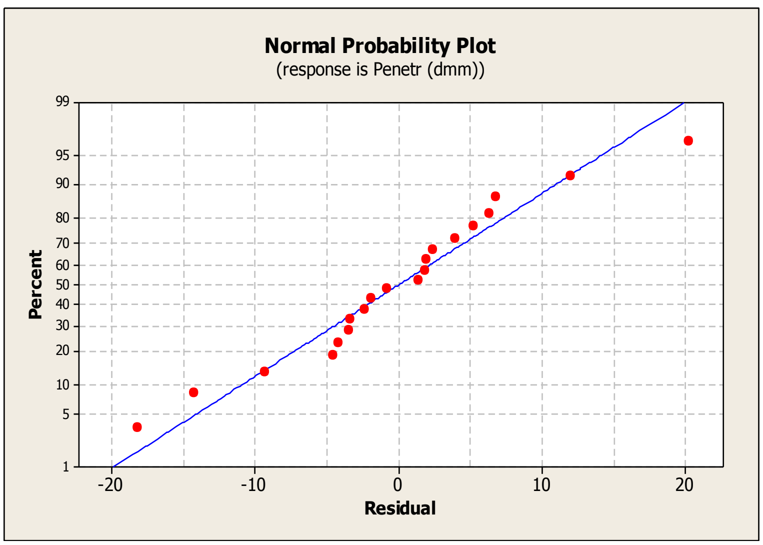


Minitab Regression model printout: PENETRATION (dmm)

Sulfur-CKD

```
Penetr (dmm)  =  47.796 + 369.96 % Sulfur - 1030 Sulfur.Sq


Coefficients

Term            Coef  SE Coef        T      P
Constant       47.80    3.942  12.1259  0.000
% Sulfur      369.96   63.299   5.8446  0.000
Sulfur.Sq   -1030.00  202.202  -5.0939  0.000

Summary of Model

S = 9.04277       R-Sq = 68.70%        R-Sq(adj) = 65.02%
PRESS = 1902.26  R-Sq(pred) = 57.17%
```

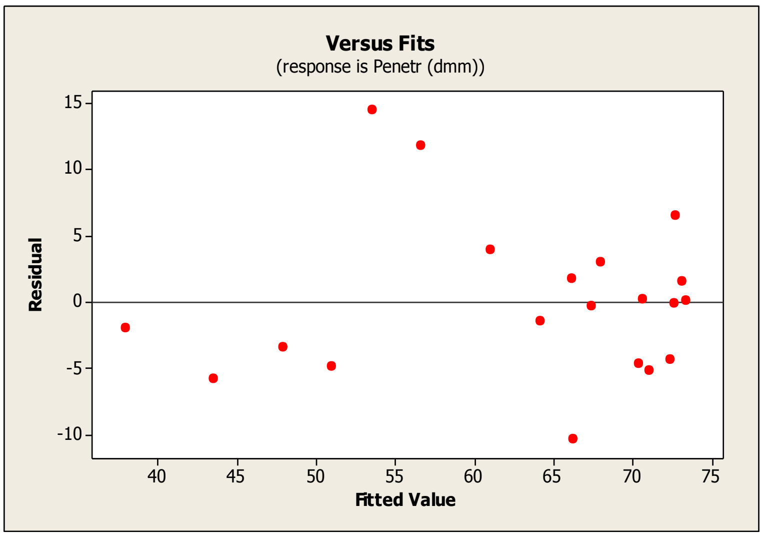


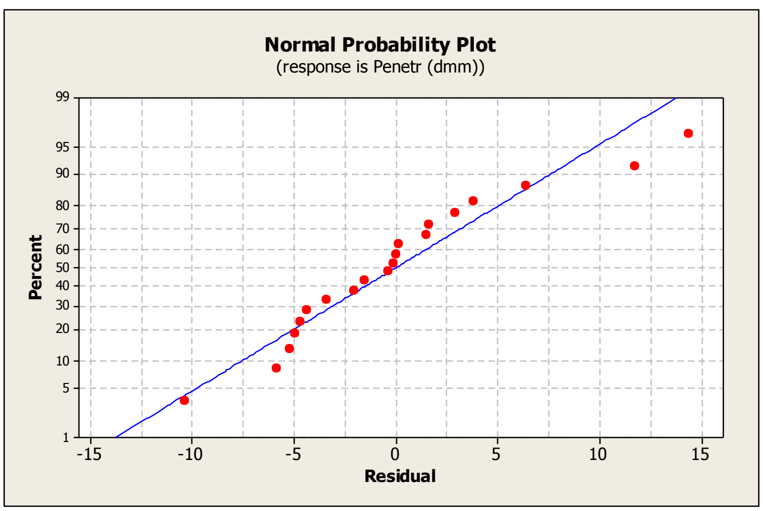


Minitab Regression model printout: PENETRATION (dmm)

Sulfur-LMD

```
Penetr (dmm)  =  53.517 + 166.615 % Sulfur + 239.393 Sulfur*LMD - 249.406
LMD.Sq
                - 394.5 Sulfur.Sq


Coefficients

Term             Coef  SE Coef        T      P
Constant       53.517    4.037  13.2559  0.000
% Sulfur      166.615   50.775   3.2814  0.005
Sulfur*LMD    239.393  143.373   1.6697  0.116
LMD.Sq       -249.406  103.866  -2.4012  0.030
Sulfur.Sq    -394.500  148.983  -2.6480  0.018

Summary of Model

S = 6.66273       R-Sq = 77.10%        R-Sq(adj) = 71.00%
PRESS = 1371.30   R-Sq(pred) = 52.84%
```





Minitab Regression model printout: PENETRATION (dmm)

Sludge-HOFA

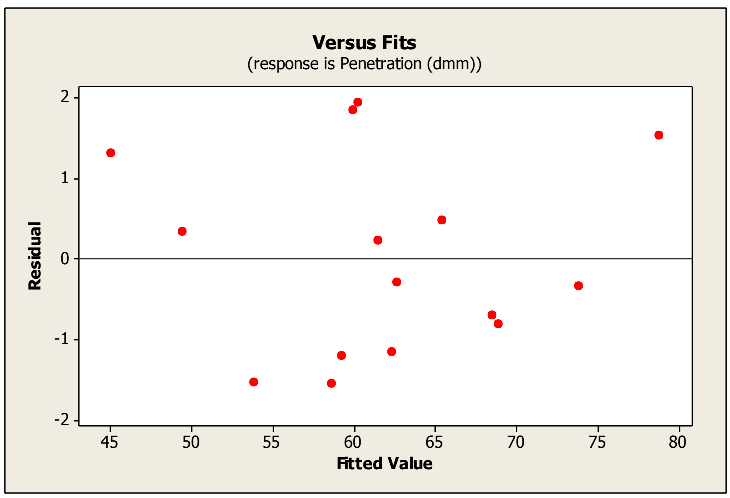
```
Penetration (dmm)  =  53.8395 - 21.8514 % Sludge + 151.748 % HOFA + 186.081
                      Sludge*HOFA - 1265.66 HOFA.sq + 3599.16 HOFA cube
```

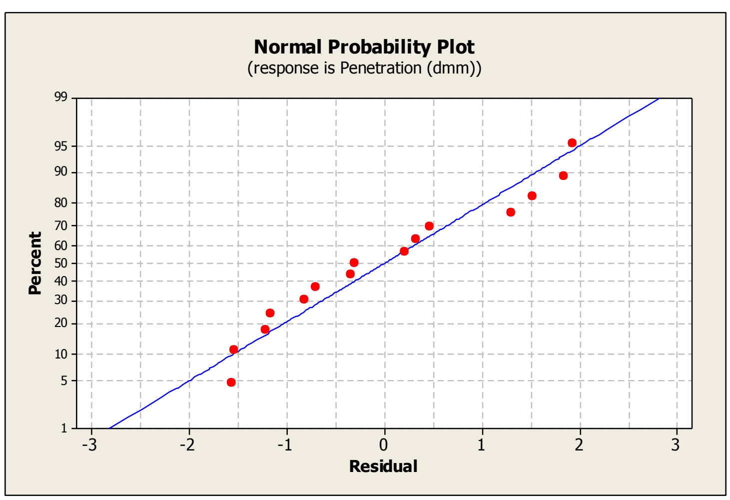
Coefficients

Term	Coef	SE Coef	T	P
Constant	53.84	1.26	42.7505	0.000
% Sludge	-21.85	4.55	-4.7988	0.001
% HOFA	151.75	41.25	3.6788	0.005
Sludge*HOFA	186.08	27.71	6.7149	0.000
HOFA.sq	-1265.66	426.25	-2.9693	0.016
HOFA cube	3599.16	1110.25	3.2418	0.010

Summary of Model

```
S = 1.50768       R-Sq = 98.10%        R-Sq(adj) = 97.05%
PRESS = 94.3453   R-Sq(pred) = 91.26%
```

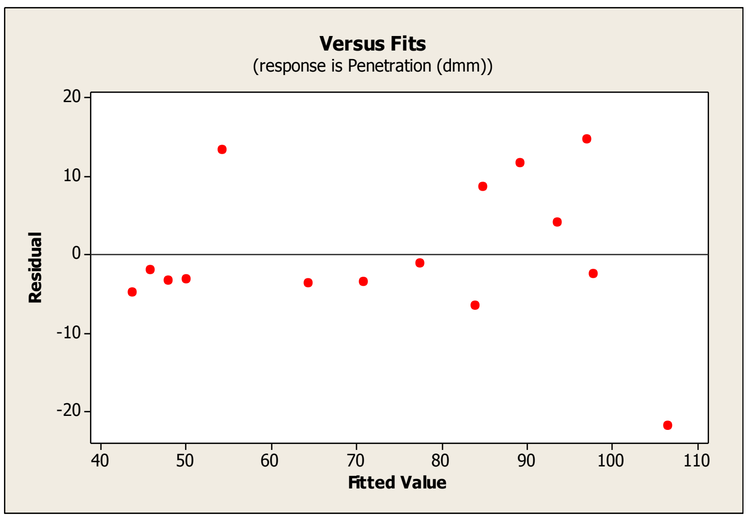


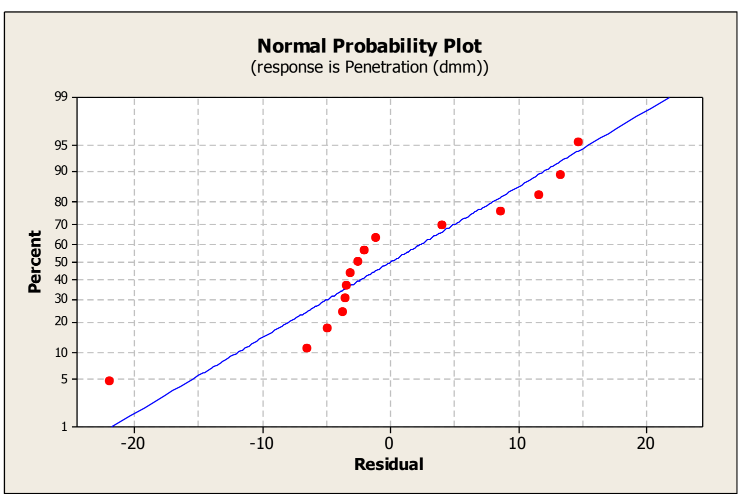


Minitab Regression model printout: PENETRATION (dmm)

Sludge-CKD

```
Penetration (dmm)  =  54.2624 + 416.073 % Sludge - 42.4459 % CKD - 220.878
                      Sludge*CKD - 772.75 Sludge.sq


Coefficients

Term            Coef  SE Coef         T      P
Constant      54.262    8.864   6.12189  0.000
% Sludge     416.073   69.222   6.01071  0.000
% CKD        -42.446   52.522  -0.80815  0.438
Sludge*CKD  -220.878  203.418  -1.08584  0.303
Sludge.sq   -772.750  151.543  -5.09921  0.000


Summary of Model

S = 11.0671       R-Sq = 84.09%        R-Sq(adj) = 77.73%
PRESS = 5002.36   R-Sq(pred) = 35.04%
```

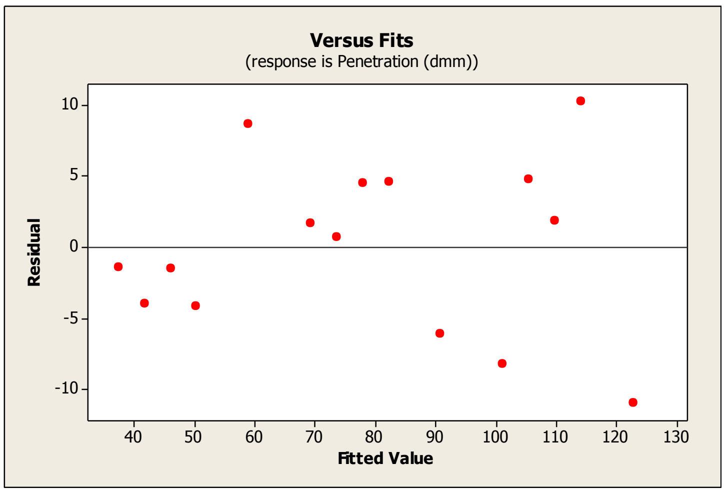


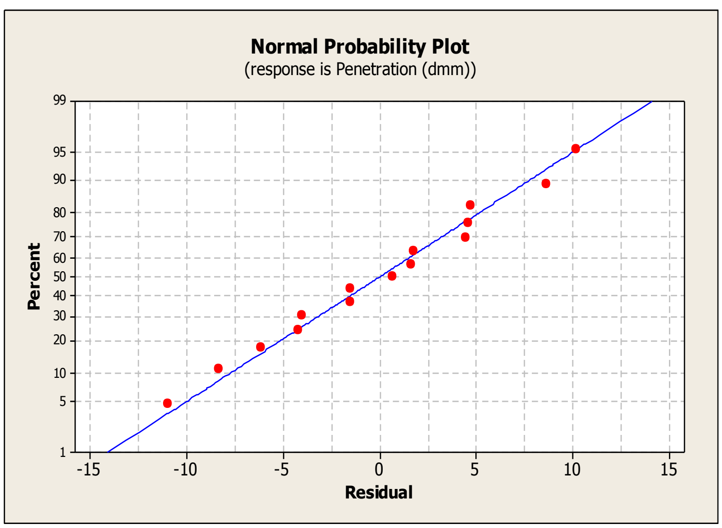


Minitab Regression model printout: PENETRATION (dmm)

Sludge-LMD

```
Penetration (dmm)  =  58.9604 + 159.5 % Sludge - 86.2883 % LMD


Coefficients

Term          Coef  SE Coef        T      P
Constant    58.960   3.8290  15.3985  0.000
% Sludge   159.500  10.3335  15.4352  0.000
% LMD      -86.288  19.6163  -4.3988  0.001

Summary of Model

S = 6.53551       R-Sq = 95.55%        R-Sq(adj) = 94.81%
PRESS = 953.409  R-Sq(pred) = 91.72%
```

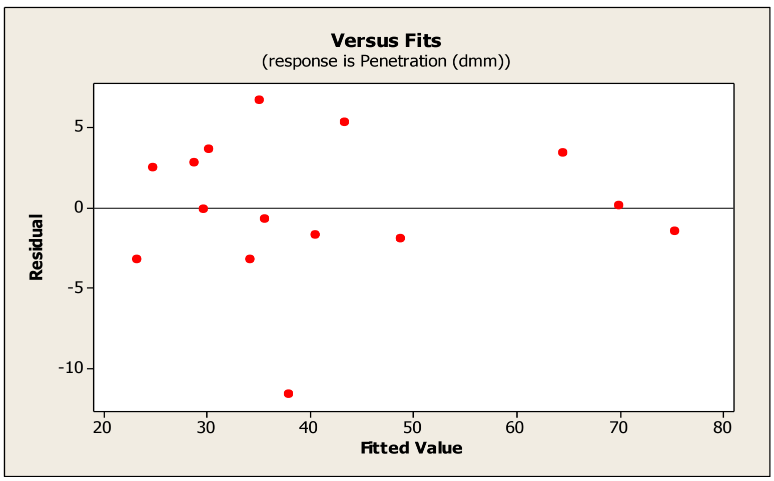


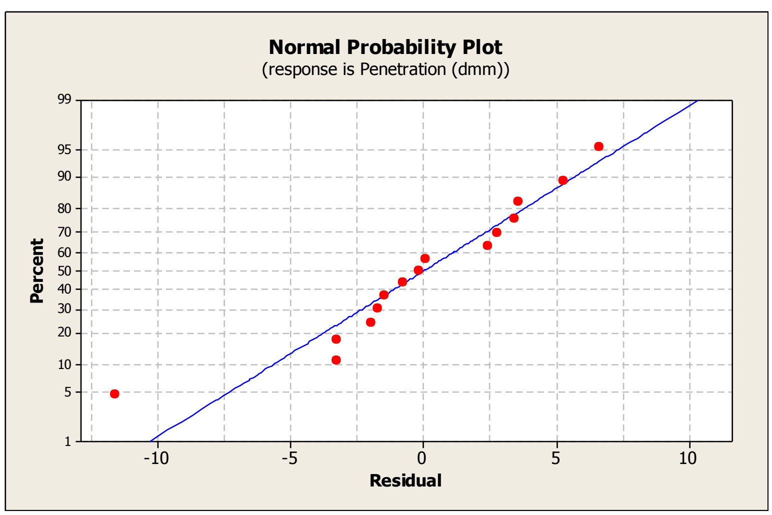


Minitab Regression model printout: PENETRATION (dmm)

Mod.Eva - HOFA

```
Penetration (dmm)  =  64.4919 + 108.8 % M.Eva - 333.588 % HOFA + 674.953
                      HOFA.sq

Coefficients

Term           Coef  SE Coef        T      P
Constant     64.492    3.253  19.8239  0.000
% M.Eva     108.800   31.616   3.4413  0.006
% HOFA     -333.588   49.211  -6.7788  0.000
HOFA.sq     674.953  190.560   3.5419  0.005

Summary of Model

S = 4.99898      R-Sq = 93.13%        R-Sq(adj) = 91.26%
PRESS = 490.295  R-Sq(pred) = 87.75%
```

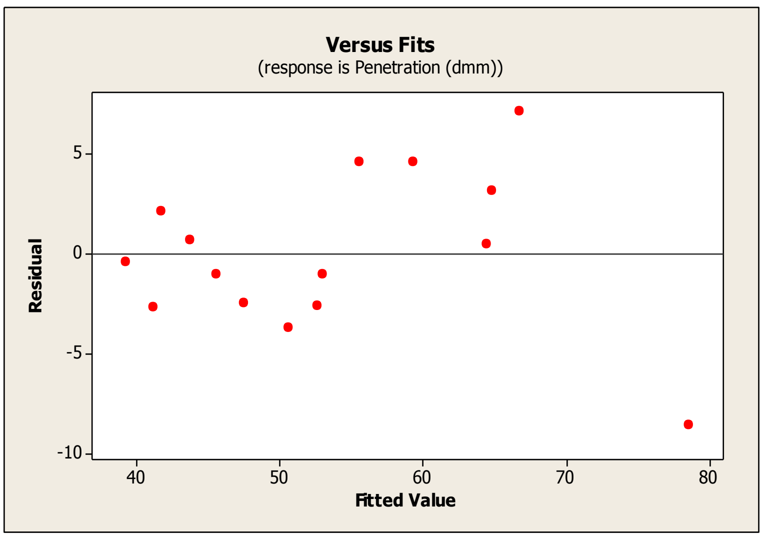


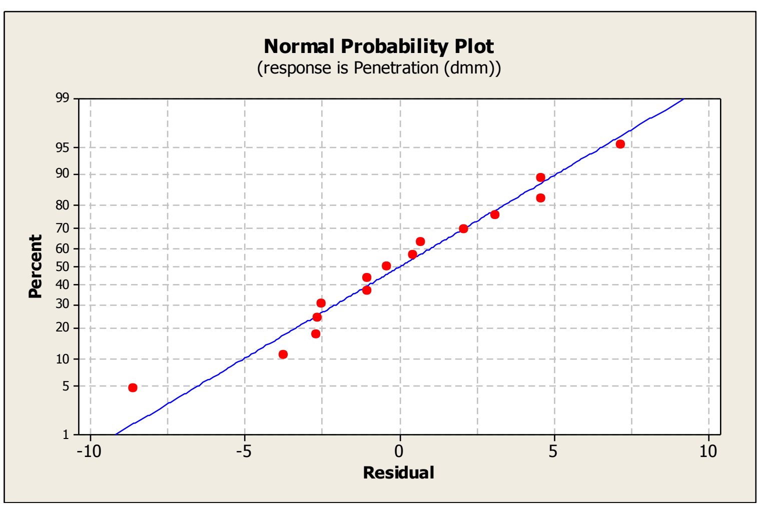


Minitab Regression model printout: PENETRATION (dmm)

Mod.Eva - CKD

```
Penetration (dmm)  =  64.7819 + 532.2 % M.Eva - 167.405 % CKD - 5124 M.Eva.sq +
                      260.55 CKD.sq

Coefficients

Term            Coef  SE Coef        T      P
Constant       64.78     3.16  20.5313  0.000
% M.Eva       532.20   106.45   4.9994  0.001
% CKD        -167.41    45.95  -3.6428  0.005
M.Eva.sq    -5124.00  1022.76  -5.0100  0.001
CKD.sq        260.55   177.95   1.4642  0.174

Summary of Model

S = 4.66823       R-Sq = 89.14%         R-Sq(adj) = 84.79%
PRESS = 619.076   R-Sq(pred) = 69.15%
```





Minitab Regression model printout: PENETRATION (dmm)

Mod.Eva - LMD

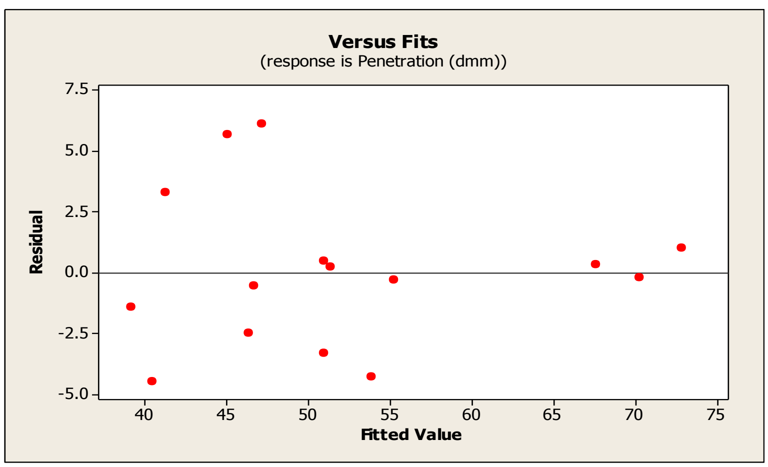
```
Penetration (dmm)  =  67.5625 + 53.1081 % M.Eva - 276.193 % LMD + 323.514
                      M.Eva*LMD + 670.29 LMD.sq
```

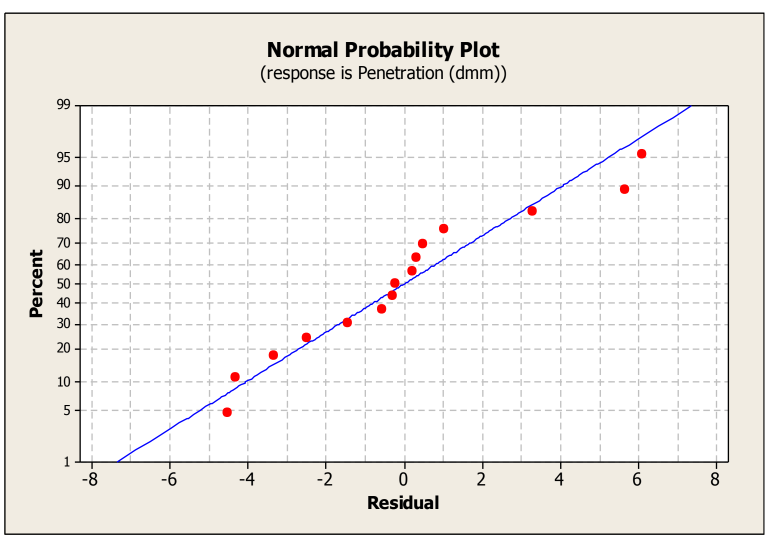
Coefficients

Term	Coef	SE Coef	T	P
Constant	67.563	3.106	21.7507	0.000
% M.Eva	53.108	45.229	1.1742	0.268
% LMD	-276.193	39.341	-7.0205	0.000
M.Eva*LMD	323.514	275.253	1.1753	0.267
LMD.sq	670.290	142.715	4.6967	0.001

Summary of Model

```
S = 3.74385       R-Sq = 91.83%        R-Sq(adj) = 88.56%
PRESS = 357.029   R-Sq(pred) = 79.19%
```

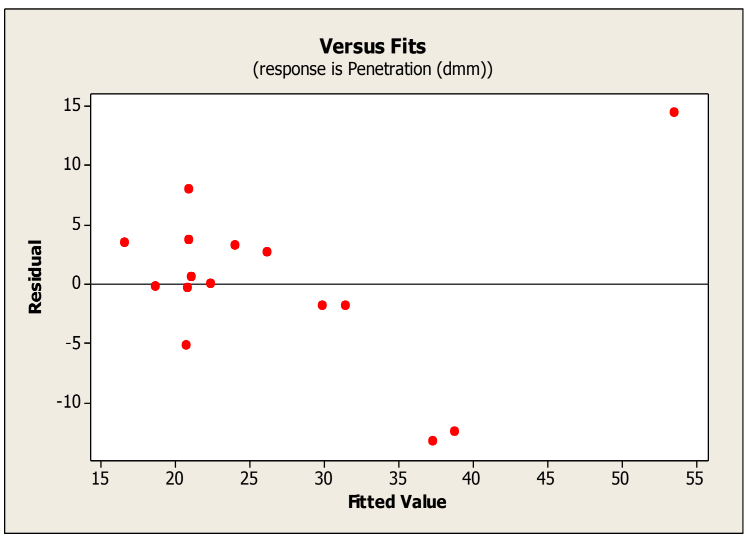


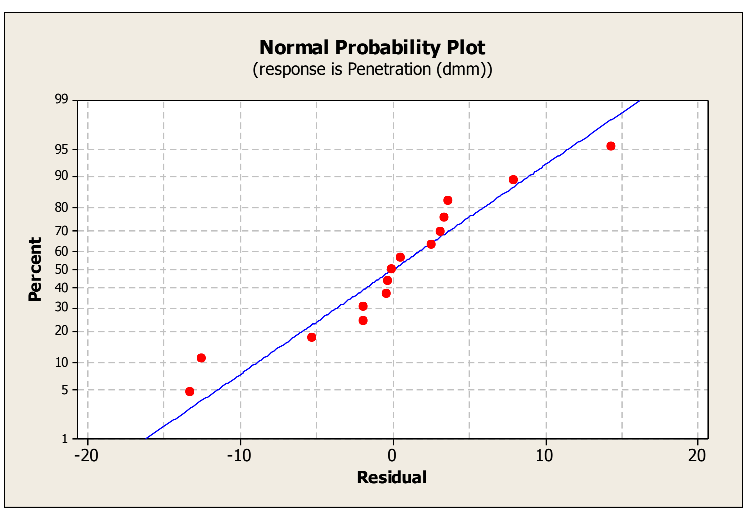


Minitab Regression model printout: PENETRATION (dmm)

SBS - HOFA

```
Penetration (dmm)  =  53.595 - 325.27 % SBS - 147.869 % HOFA + 1466.22 SBS*HOFA


Coefficients

Term           Coef  SE Coef         T      P
Constant      53.60    6.113   8.76770  0.000
% SBS       -325.27   94.699  -3.43479  0.006
% HOFA      -147.87   37.201  -3.97485  0.002
SBS*HOFA    1466.22  576.319   2.54411  0.027

Summary of Model

S = 7.83878       R-Sq = 67.04%        R-Sq(adj) = 58.05%
PRESS = 2163.90   R-Sq(pred) = -5.52%
```

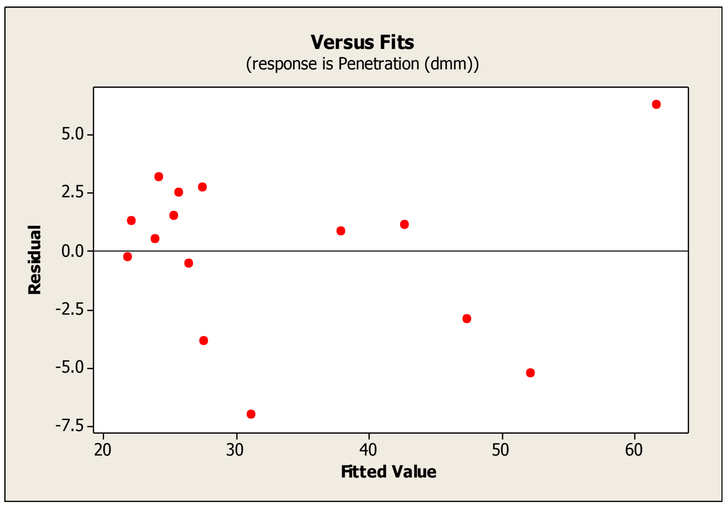


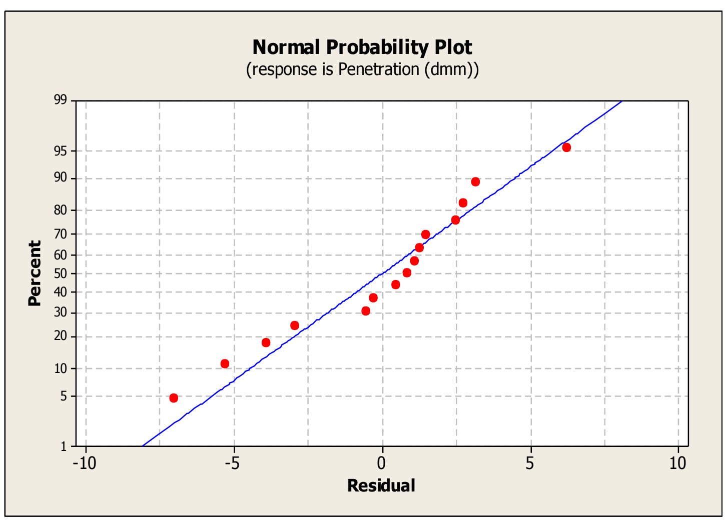


Minitab Regression model printout: PENETRATION (dmm)

SBS - CKD

```
Penetration (dmm)  =  61.6523 - 823.946 % SBS - 94.8018 % CKD + 1176.76 SBS*CKD
                      + 4260 SBS.sq


Coefficients

Term         Coef  SE Coef        T      P
Constant    61.65    3.299  18.6888  0.000
% SBS     -823.95  103.052  -7.9954  0.000
% CKD      -94.80   19.548  -4.8497  0.001
SBS*CKD   1176.76  302.833   3.8858  0.003
SBS.sq    4260.00  902.423   4.7206  0.001

Summary of Model

S = 4.11898      R-Sq = 92.65%        R-Sq(adj) = 89.70%
PRESS = 597.424  R-Sq(pred) = 74.10%
```

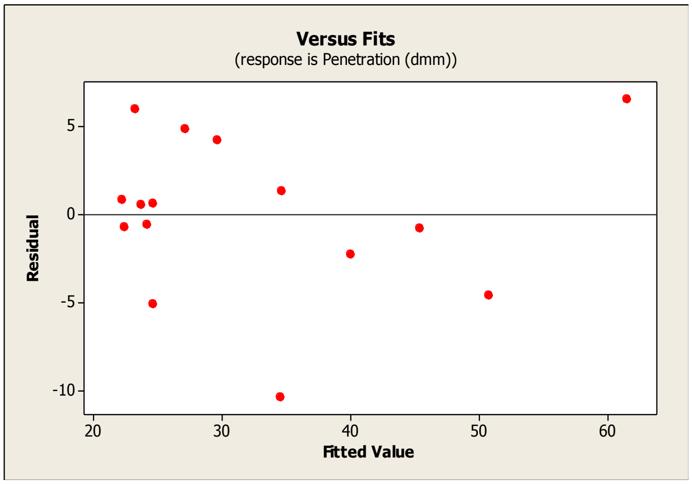


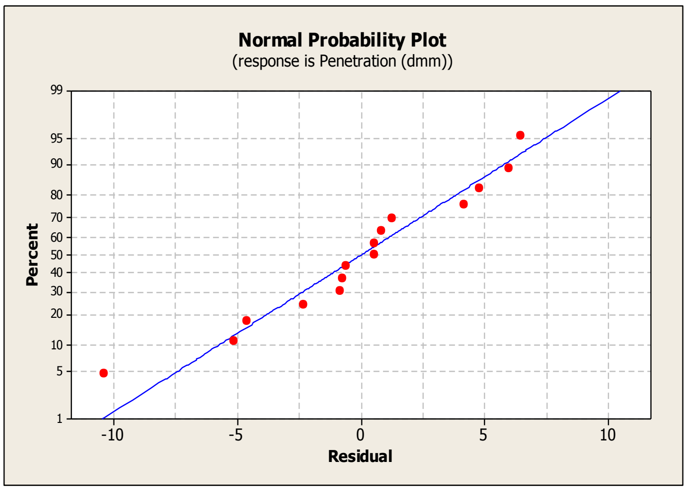


Minitab Regression model printout: PENETRATION (dmm)

SBS - LMD

```
Penetration (dmm)  =  61.4202 - 687.449 % SBS - 107.144 % LMD + 1158.92 SBS*LMD
                      + 2968 SBS.sq


Coefficients

Term          Coef  SE Coef        T      P
Constant     61.42     4.26  14.4163  0.000
% SBS      -687.45   133.09  -5.1653  0.000
% LMD      -107.14    25.25  -4.2441  0.002
SBS*LMD    1158.92   391.10   2.9632  0.014
SBS.sq     2968.00  1165.46   2.5466  0.029

Summary of Model

S = 5.31959      R-Sq = 87.63%        R-Sq(adj) = 82.68%
PRESS = 884.450  R-Sq(pred) = 61.34%
```

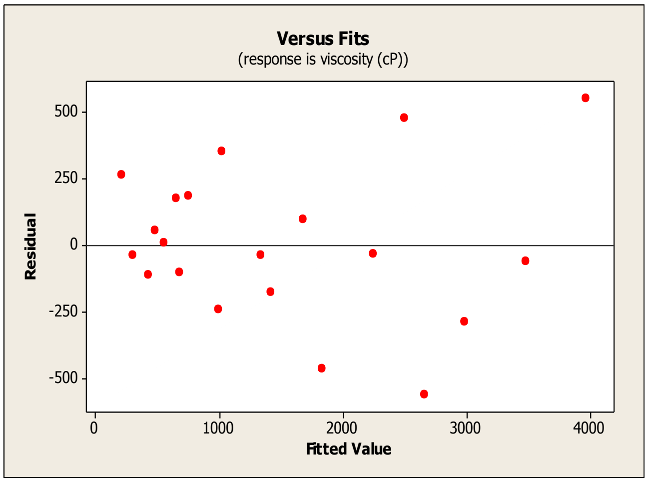


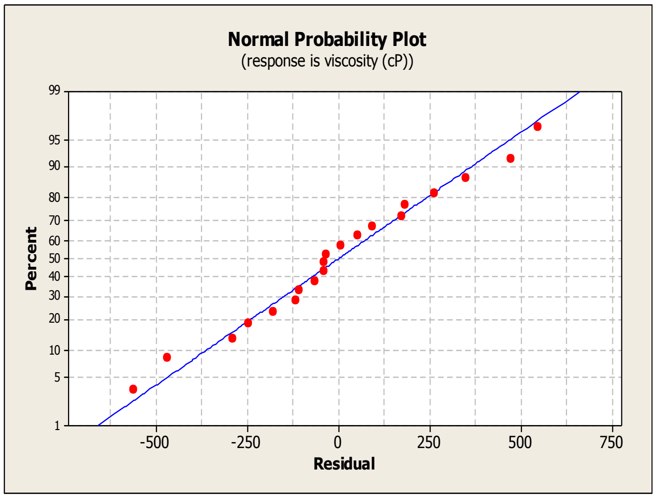


Minitab Regression model printout: VISCOSITY (cP)

Sulfur-HOFA

```
viscosity (cP)  =  680.75 - 1263.2 % Sulfur - 3008.94 % HOFA - 14477.8
                   Sulfur*HOFA + 64590.4 HOFA.Sq

Coefficients

Term              Coef  SE Coef         T      P
Constant         680.8    241.7   2.81683  0.013
% Sulfur       -1263.2   1221.8  -1.03389  0.318
% HOFA         -3008.9   2945.8  -1.02142  0.323
Sulfur*HOFA   -14477.8   7435.6  -1.94709  0.070
HOFA.Sq        64590.4  10558.1   6.11764  0.000

Summary of Model

S = 319.818       R-Sq = 93.96%        R-Sq(adj) = 92.35%
PRESS = 3288785   R-Sq(pred) = 87.06%
```

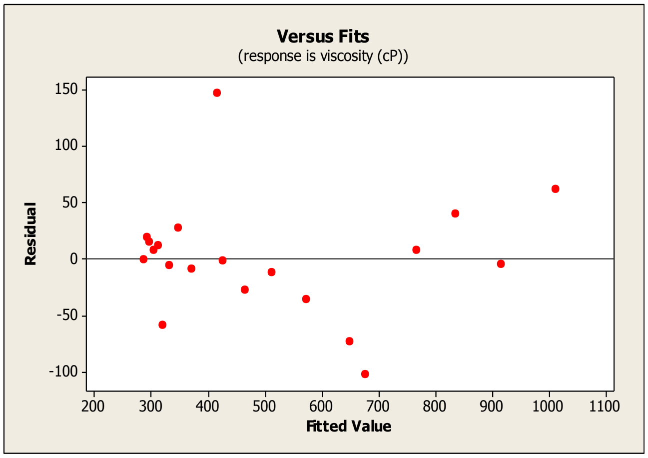


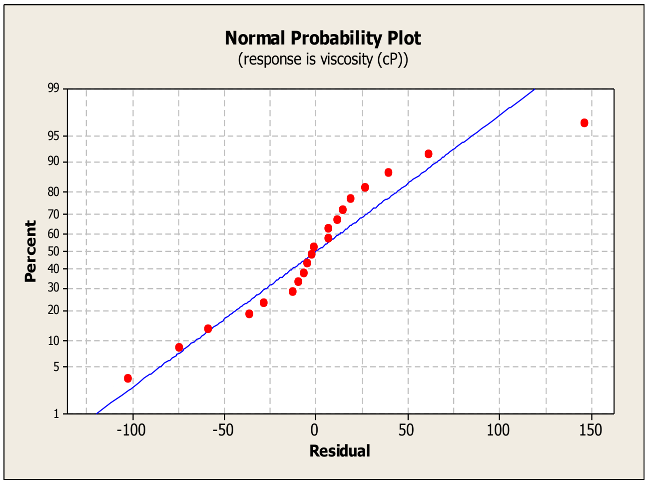


Minitab Regression model printout: VISCOSITY (cP)

Sulfur-CKD

```
viscosity (cP)  =  677.403 - 3325 % Sulfur + 614.562 % CKD - 4125 Sulfur*CKD +
                   2918.81 CKD.Sq + 7125 Sulfur.Sq

Coefficients

Term            Coef  SE Coef        T      P
Constant      677.40    47.16  14.3638  0.000
% Sulfur    -3325.00   461.99  -7.1971  0.000
% CKD         614.56   551.23   1.1149  0.284
Sulfur*CKD  -4125.00  1391.37  -2.9647  0.010
CKD.Sq       2918.81  1975.64   1.4774  0.162
Sulfur.Sq    7125.00  1338.17   5.3244  0.000

Summary of Model

S = 59.8450      R-Sq = 95.20%        R-Sq(adj) = 93.49%
PRESS = 180852   R-Sq(pred) = 82.70%
```

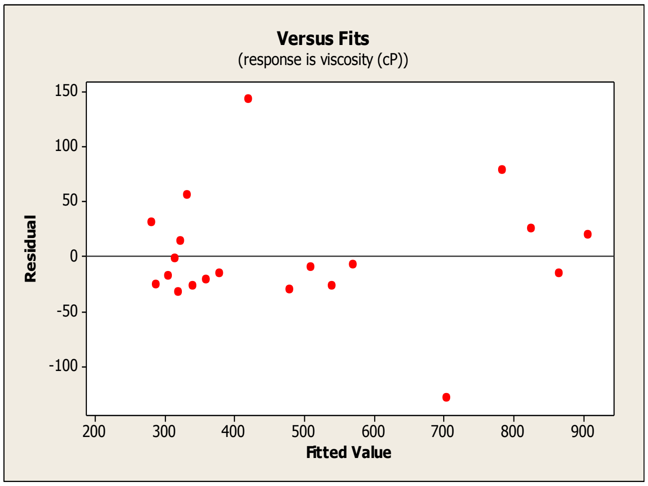


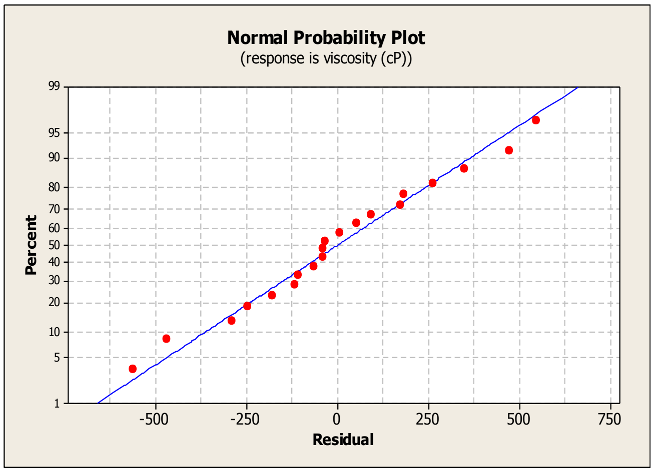


Minitab Regression model printout: VISCOSITY (cP)

Sulfur-LMD

```
viscosity (cP)  =  703.581 - 3558.45 % Sulfur + 810.135 % LMD - 2118.24
                   Sulfur*LMD + 7250 Sulfur.Sq

Coefficients

Term            Coef  SE Coef        T      P
Constant      703.58    44.79  15.7092  0.000
% Sulfur    -3558.45   461.69  -7.7074  0.000
% LMD         810.14   260.14   3.1143  0.007
Sulfur*LMD  -2118.24  1390.48  -1.5234  0.148
Sulfur.Sq    7250.00  1337.32   5.4213  0.000

Summary of Model

S = 59.8070      R-Sq = 94.09%         R-Sq(adj) = 92.52%
PRESS = 157073  R-Sq(pred) = 82.71%
```

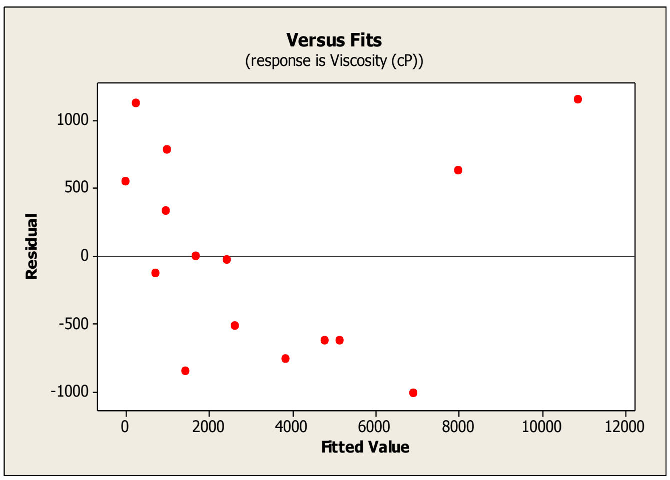


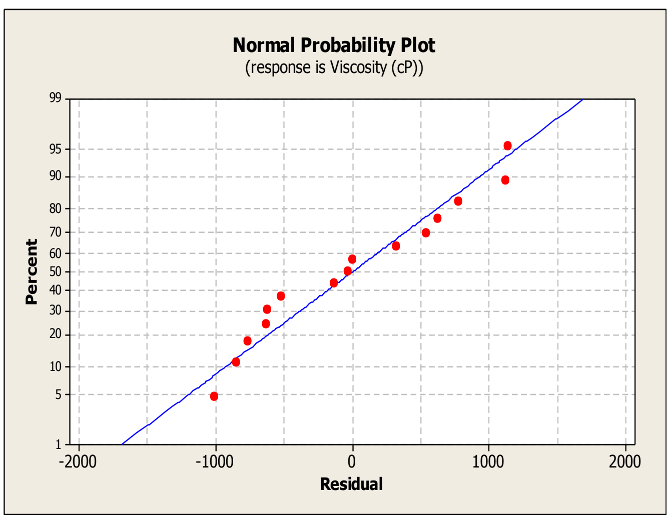


Minitab Regression model printout: VISCOSITY (cP)

Sludge-HOFA

```
Viscosity (cP)  =  1422.91 - 3599.73 % Sludge - 29439.2 % HOFA + 71608.8
                   Sludge*HOFA + 177136 HOFA.sq


Coefficients

Term            Coef  SE Coef         T      P
Constant        1423    712.4   1.99747  0.074
% Sludge       -3600   2593.1  -1.38820  0.195
% HOFA        -29439   9022.1  -3.26300  0.009
Sludge*HOFA    71609  15781.1   4.53763  0.001
HOFA.sq       177136  32729.1   5.41220  0.000


Summary of Model

S = 858.585       R-Sq = 95.10%        R-Sq(adj) = 93.14%
PRESS = 26781192  R-Sq(pred) = 82.20%
```

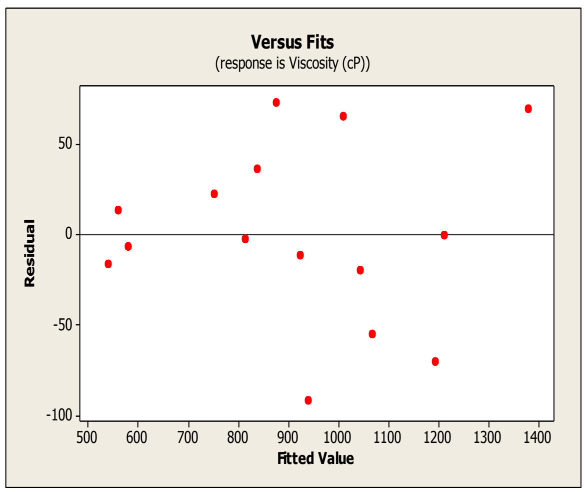


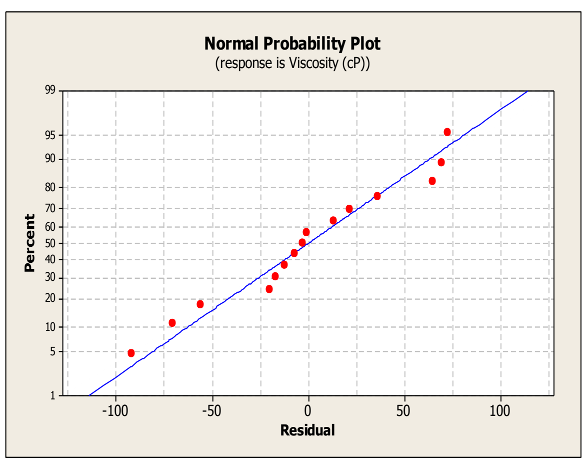


Minitab Regression model printout: VISCOSITY (cP)

Sludge-CKD

```
Viscosity (cP)  =  581.863 - 99.6284 % Sludge + 1713.24 % CKD + 4102.7
                   Sludge*CKD


Coefficients

Term            Coef  SE Coef        T      P
Constant      581.86    43.13  13.4899  0.000
% Sludge      -99.63   167.05  -0.5964  0.563
% CKD        1713.24   262.50   6.5266  0.000
Sludge*CKD   4102.70  1016.66   4.0355  0.002

Summary of Model

S = 55.3123      R-Sq = 96.20%        R-Sq(adj) = 95.17%
PRESS = 63792.4  R-Sq(pred) = 92.80%
```

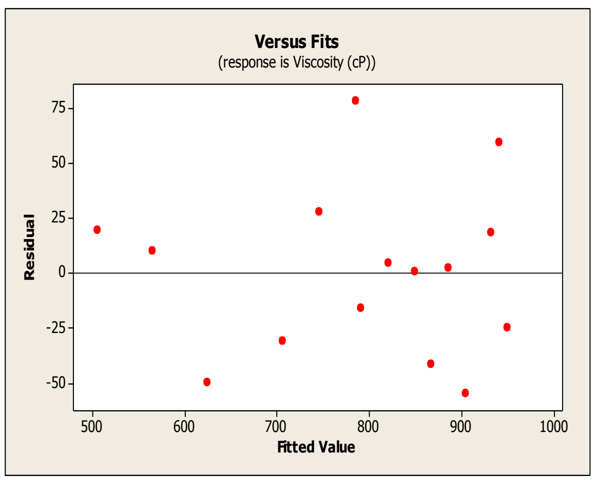


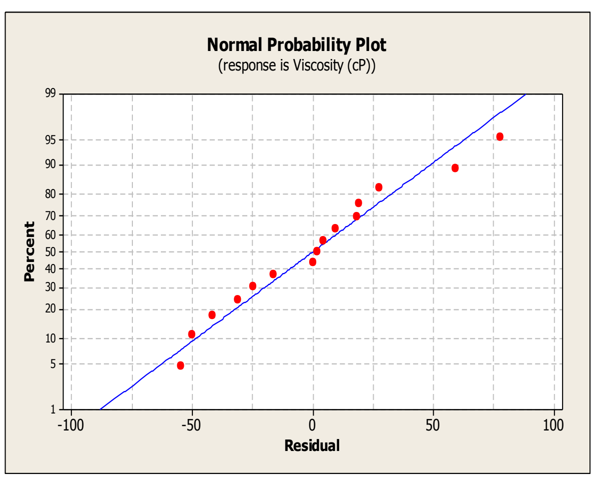


Minitab Regression model printout: VISCOSITY (cP)

Sludge-LMD

```
Viscosity (cP)  =  625.013 - 298.142 % Sludge + 1796.34 % LMD + 1013.51
                   Sludge*LMD - 1989.49 LMD.sq


Coefficients

Term            Coef  SE Coef        T      P
Constant      625.01    37.28  16.7669  0.000
% Sludge     -298.14   135.69  -2.1972  0.053
% LMD        1796.34   472.11   3.8049  0.003
Sludge*LMD   1013.51   825.80   1.2273  0.248
LMD.sq      -1989.49  1712.66  -1.1616  0.272

Summary of Model

S = 44.9285       R-Sq = 93.01%         R-Sq(adj) = 90.21%
PRESS = 64437.9   R-Sq(pred) = 77.68%
```

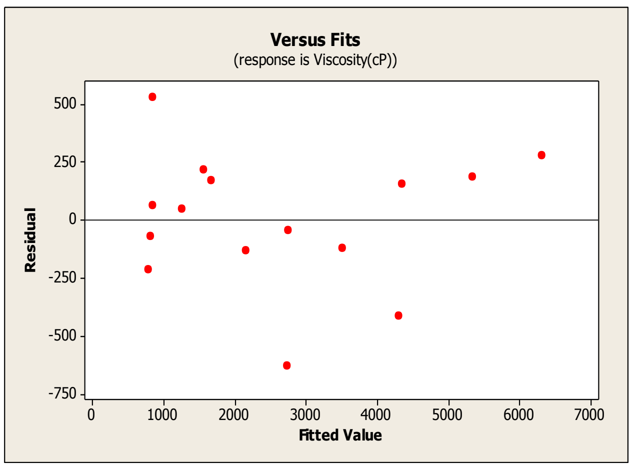


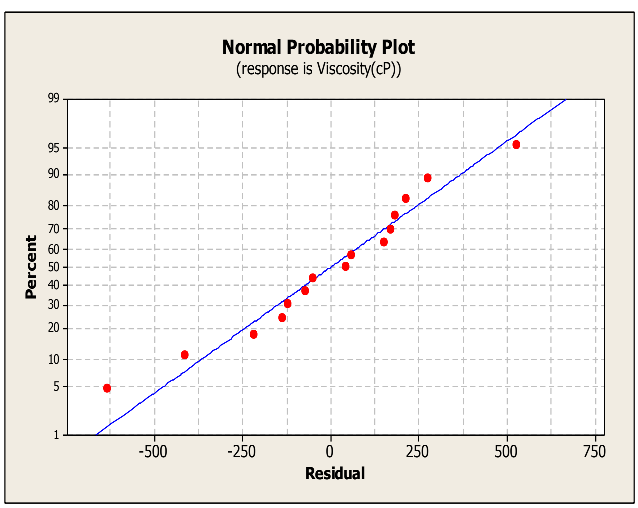


Minitab Regression model printout: VISCOSITY (cP)

Mod.Eva - HOFA

```
Viscosity(cP)  =  790.986 + 606.081 % M.Eva - 8606.4 % HOFA + 76035.1
                  M.Eva*HOFA + 91530.6 HOFA.sq

Coefficients

Term            Coef  SE Coef         T      P
Constant       791.0    281.5   2.81016  0.018
% M.Eva        606.1   4098.4   0.14788  0.885
% HOFA       -8606.4   3564.9  -2.41420  0.036
M.Eva*HOFA   76035.1  24942.4   3.04843  0.012
HOFA.sq      91530.6  12932.2   7.07770  0.000

Summary of Model

S = 339.253      R-Sq = 97.50%        R-Sq(adj) = 96.49%
PRESS = 2814213  R-Sq(pred) = 93.88%
```

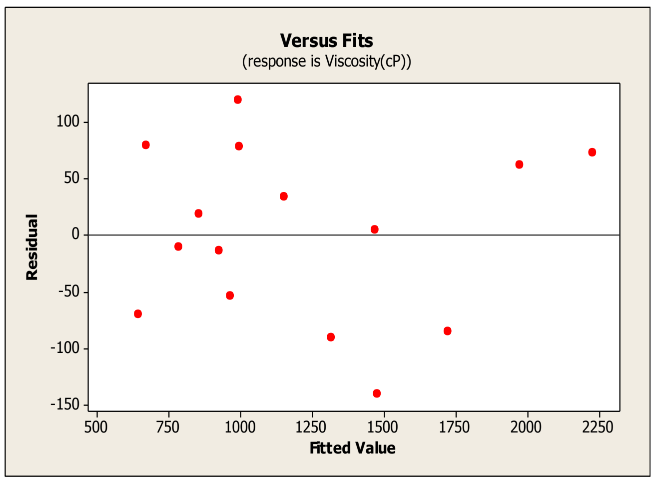


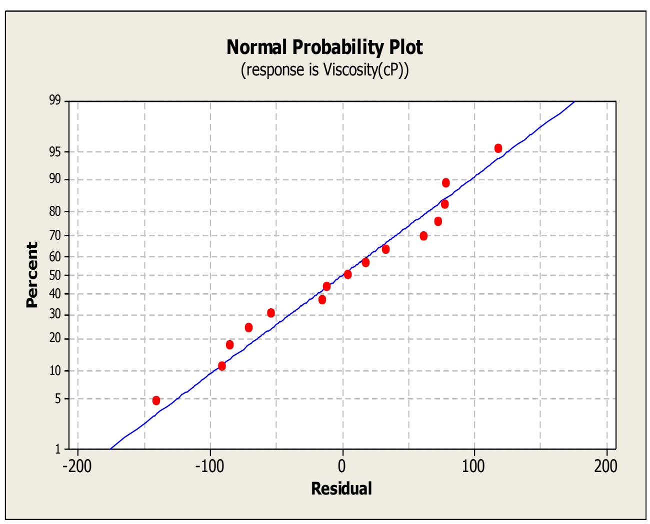


Minitab Regression model printout: VISCOSITY (cP)

Mod.Eva - CKD

```
Viscosity(cP)  =  645.529 - 2196.97 % M.Eva + 1406.94 % CKD + 36378.4 M.Eva*CKD
                  + 54040 M.Eva.sq

Coefficients

Term            Coef  SE Coef         T      P
Constant       645.5     71.8   8.99441  0.000
% M.Eva      -2197.0   2242.0  -0.97992  0.350
% CKD         1406.9    425.3   3.30827  0.008
M.Eva*CKD    36378.4   6588.4   5.52160  0.000
M.Eva.sq     54040.0  19632.9   2.75252  0.020

Summary of Model

S = 89.6117      R-Sq = 97.54%         R-Sq(adj) = 96.55%
PRESS = 217640   R-Sq(pred) = 93.33%
```

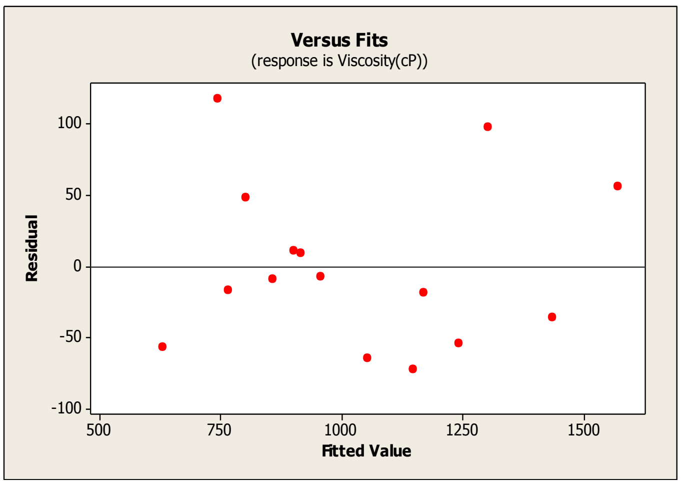


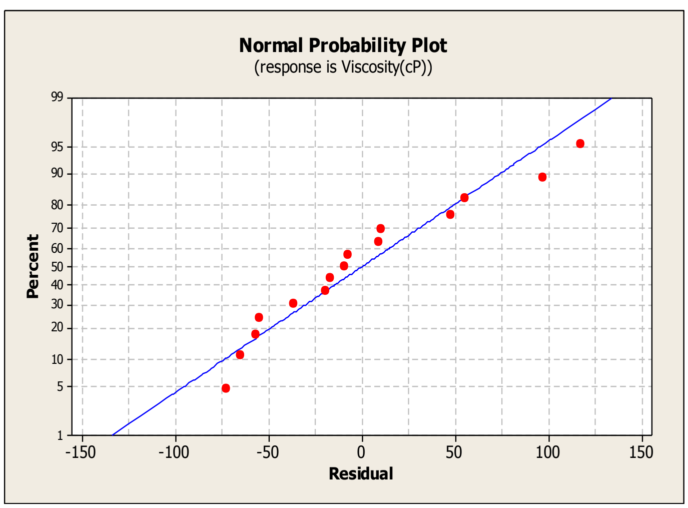


Minitab Regression model printout: VISCOSITY (cP)

Mod.Eva - LMD

```
Viscosity(cP)  =  631.721 + 2702.7 % M.Eva + 1136.76 % LMD + 15337.8 M.Eva*LMD


Coefficients

Term            Coef  SE Coef        T      P
Constant       631.7    50.72  12.4558  0.000
% M.Eva       2702.7   785.71   3.4398  0.006
% LMD         1136.8   308.65   3.6829  0.004
M.Eva*LMD    15337.8  4781.65   3.2076  0.008

Summary of Model

S = 65.0375       R-Sq = 95.69%         R-Sq(adj) = 94.52%
PRESS = 89625.9   R-Sq(pred) = 91.70%
```





APPENDIX C

Overview of Analysis of variance (ANOVA)

Analysis of Variance (ANOVA) [41], is a parametric procedure that yields values that can be used to determine whether a statistically significant relation exists between *dependent variable* (X) and *independent variables* (Y). By parametric, it is meant that the data are *normally* distributed in a *normal* or *bell-shaped* curve. The dependent variable may be referred to as the "*response*" or "*outcome variable*". Independent variables are sometimes called "factors" or "predictors". The ANOVA model is a univariate model, in that interest is in how the predictors affect a single dependent variable. A one-way ANOVA compares the means of a variable that is classified by only one variable, which is called the *factor*. The possible *values* of the factor are called the *levels* of the factor. In the two-way ANOVA model, there are **two factors**, each with its own number of levels. When one is interested in the effects of two factors, it is much more advantageous to perform a two-way analysis of variance, as opposed to two separate one-way ANOVAs.

In ANOVA, the following statistical terminologies are used:

Data points

Data points are the replicate observations of the dependent variable (X_1, X_2, X_i, X_n) measured at each level of the independent variable.

Sample mean $\left(\overline{X}\right)$ $\overline{X}$ = total sum of data points/total number of data points

Correction factor (CF): CF = (total sum of all data points)^2/total number of data points

Degree of freedom (df):

Degree of freedom is the number of values in the final calculation of a statistic that are free to vary.

df $= n - 1$

where n represents the number of groups

Error (residual)

It is the amount by which an observed variate differs from the value predicted by the assumed statistical model. Errors or residuals are the segments of scores not accounted for by the analysis. In ANOVA, the errors are assumed to be independent of each other, and are assumed *normally* distributed about the sample means. They are also assumed to be identically distributed for each sample (since the analysis is seeking only a significant difference between sample means), which is known as the assumption of homogeneity of variances.

Sum of squares (SS)

The sum of square is the squared distance between each data point (X_i) and the sample mean**(** $\overline{X}$ **)**, summed for all N data points.

$$\text{SS} = \sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2$$

where X_i represents the i observations and $\overline{X}$ represents the sample mean.

Mean square (MS)

Mean square is a measure of the variability of *group mean* around the *grand mean*. It is the average sum of squares. In other words MS is the sum of squared deviations from the mean divided by the appropriate degrees of freedom.

Null hypothesis

The hypothesis used in statistics to propose that no statistical significance exists in a set of given observations is called a *Null* hypothesis, H_o. The *null* hypothesis is presumed to be true until statistical evidence (through testing of a hypothesis) *nullifies* it for an *alternative* hypothesis, H_a.

F-ratio

The ratio is a statistical measure calculated by procedure of variance analysis, and reveals the significance of a hypothesis that *dependent* variable depends on *independent* variable. It comprises the ratio of two mean- squares. The F-ratio tells us precisely how much more of the variation in *Y* is explained by *X*. A large proportion indicates a significant effect of *Y*. The observed F-ratio is connected by an equation to the exact probability of a true null hypothesis, (i.e. that the ratio equals unity), but the standard tables can be used to find out whether the observed F-ratio indicates a significant relationship.

F-ratio = MS of the source effect/ MS of the residual error.

P-value

P-value is a measure of *acceptance* or *rejection* of a statistical significance based on a standard that no more than 5 % (0.05 level) of the difference is due to chance or sampling error.

APPENDIX D

MINITAB PRINTOUT OF RESULTS ANALYSIS OF VARIANCE (ANOVA)

Minitab ANOVA printout: SOFTENING POINT

Two-way ANOVA: softening point versus % sulfur, %HOFA

Source	DF	SS	MS	F	P
% sulfur	3	125.186	41.729	8.67	0.002
%HOFA	4	493.865	123.466	25.64	0.000
Error	12	57.787	4.816		
Total	19	676.837			

S = 2.194   R-Sq = 91.46%   R-Sq(adj) = 86.48%

Two-way ANOVA: softening point versus % sulfur, %CKD

Source	DF	SS	MS	F	P
% sulfur	3	66.577	22.1925	4.44	0.026
%CKD	4	37.318	9.3295	1.87	0.181
Error	12	59.990	4.9992		
Total	19	163.885			

S = 2.236   R-Sq = 63.40%   R-Sq(adj) = 42.04%

Two-way ANOVA: softening point versus % sulfur, %LMD

Source	DF	SS	MS	F	P
% sulfur	3	194.266	64.7553	13.33	0.000
%LMD	4	19.613	4.9033	1.01	0.440
Error	12	58.279	4.8566		
Total	19	272.158			

S = 2.204   R-Sq = 78.59%   R-Sq(adj) = 66.10%

Two-way ANOVA: Softening point (oC) versus % Sludge, % HOFA

Source	DF	SS	MS	F	P
% Sludge	2	7.14	3.571	0.24	0.795
% HOFA	4	951.66	237.914	15.76	0.001
Error	8	120.75	15.094		
Total	14	1079.55			

S = 3.885   R-Sq = 88.81%   R-Sq(adj) = 80.43%

Two-way ANOVA: Softening point (oC) versus % Sludge, % CKD

Source	DF	SS	MS	F	P
% Sludge	2	2.305	1.1527	0.32	0.736
% CKD	4	100.916	25.2290	6.96	0.010
Error	8	28.988	3.6235		
Total	14	132.209			

S = 1.904   R-Sq = 78.07%   R-Sq(adj) = 61.63%

Minitab ANOVA printout: SOFTENING POINT

Two-way ANOVA: Softening point (oC) versus % Sludge, % LMD

Source	DF	SS	MS	F	P
% Sludge	2	126.241	63.1207	61.99	0.000
% LMD	4	41.031	10.2577	10.07	0.003
Error	8	8.145	1.0182		
Total	14	175.417			

S = 1.009   R-Sq = 95.36%   R-Sq(adj) = 91.87%

Two-way ANOVA: Softening point (oC) versus % SBS, % HOFA

Source	DF	SS	MS	F	P
% SBS	2	3507.65	1753.82	54.08	0.000
% HOFA	4	90.52	22.63	0.70	0.615
Error	8	259.46	32.43		
Total	14	3857.63			

S = 5.695   R-Sq = 93.27%   R-Sq(adj) = 88.23%

Two-way ANOVA: Softening point (oC) versus % SBS, % CKD

Source	DF	SS	MS	F	P
% SBS	2	4870.54	2435.27	144.81	0.000
% CKD	4	126.04	31.51	1.87	0.209
Error	8	134.54	16.82		
Total	14	5131.12			

S = 4.101   R-Sq = 97.38%   R-Sq(adj) = 95.41%

Two-way ANOVA: Softening point (oC) versus % SBS, % LMD

Source	DF	SS	MS	F	P
% SBS	2	4234.02	2117.01	105.83	0.000
% LMD	4	60.04	15.01	0.75	0.585
Error	8	160.03	20.00		
Total	14	4454.09			

S = 4.473   R-Sq = 96.41%   R-Sq(adj) = 93.71%

Two-way ANOVA: Softening point (oC) versus % M.Eva, % HOFA

Source	DF	SS	MS	F	P
% M.Eva	2	10.228	5.114	1.24	0.338
% HOFA	4	302.416	75.604	18.40	0.000
Error	8	32.872	4.109		
Total	14	345.516			

S = 2.027   R-Sq = 90.49%   R-Sq(adj) = 83.35%

Minitab ANOVA printout: SOFTENING POINT

Two-way ANOVA: Softening point (oC) versus % M.Eva, % CKD

```
Source   DF       SS       MS      F      P
% M.Eva   2  113.585  56.7927  52.50  0.000
% CKD     4   54.769  13.6923  12.66  0.002
Error     8    8.655   1.0818
Total    14  177.009
S = 1.040   R-Sq = 95.11%   R-Sq(adj) = 91.44%
```

Two-way ANOVA: Softening point (oC) versus % M.Eva, % LMD

```
Source   DF       SS       MS      F      P
% M.Eva   2  30.8253  15.4127  12.21  0.004
% LMD     4  43.1507  10.7877   8.54  0.005
Error     8  10.1013   1.2627
Total    14  84.0773

S = 1.124   R-Sq = 87.99%   R-Sq(adj) = 78.97%
```

Minitab ANOVA printout: DUCTILITY

Two-way ANOVA: Ductility (Cm) versus % sulfur, %HOFA

Source	DF	SS	MS	F	P
% sulfur	3	397.9	132.63	0.33	0.803
%HOFA	4	19026.7	4756.66	11.90	0.000
Error	12	4795.7	399.64		
Total	19	24220.3			

S = 19.99   R-Sq = 80.20%   R-Sq(adj) = 68.65%

Two-way ANOVA: Ductility (Cm) versus % Sulfur, % CKD

Source	DF	SS	MS	F	P
% Sulfur	3	4089.8	1363.28	6.20	0.009
% CKD	4	13753.5	3438.36	15.63	0.000
Error	12	2639.9	219.99		
Total	19	20483.1			

S = 14.83   R-Sq = 87.11%   R-Sq(adj) = 79.59%

Two-way ANOVA: Ductility (Cm) versus % sulfur, %LMD

Source	DF	SS	MS	F	P
% sulfur	3	4354.7	1451.57	4.26	0.029
%LMD	4	6498.4	1624.61	4.76	0.016
Error	12	4091.5	340.96		
Total	19	14944.7			

S = 18.47   R-Sq = 72.62%   R-Sq(adj) = 56.65%

Two-way ANOVA: Ductility (cm) versus % Sludge, % HOFA

Source	DF	SS	MS	F	P
% Sludge	2	2813.2	1406.59	1.72	0.239
% HOFA	4	9217.8	2304.44	2.82	0.099
Error	8	6532.7	816.59		
Total	14	18563.6			

S = 28.58   R-Sq = 64.81%   R-Sq(adj) = 38.42%

Two-way ANOVA: Ductility (cm) versus % Sludge, % CKD

Source	DF	SS	MS	F	P
% Sludge	2	6605.1	3302.55	5.84	0.027
% CKD	4	4905.9	1226.47	2.17	0.163
Error	8	4524.2	565.52		
Total	14	16035.1			

S = 23.78   R-Sq = 71.79%   R-Sq(adj) = 50.63%

Minitab ANOVA printout: DUCTILITY

Two-way ANOVA: Ductility (cm) versus % Sludge, % LMD

Source	DF	SS	MS	F	P
% Sludge	2	9476.1	4738.04	7.96	0.013
% LMD	4	3088.3	772.08	1.30	0.349
Error	8	4764.3	595.54		
Total	14	17328.7			

S = 24.40   R-Sq = 72.51%   R-Sq(adj) = 51.89%

Two-way ANOVA: Ductility (cm) versus % SBS, % HOFA

Source	DF	SS	MS	F	P
% SBS	2	5104.7	2552.33	2.35	0.158
% HOFA	4	5875.2	1468.79	1.35	0.332
Error	8	8703.0	1087.87		
Total	14	19682.8			

S = 32.98   R-Sq = 55.78%   R-Sq(adj) = 22.62%

Two-way ANOVA: Ductility (cm) versus % SBS, % CKD

Source	DF	SS	MS	F	P
% SBS	2	12111.8	6055.91	8.69	0.010
% CKD	4	3548.1	887.03	1.27	0.356
Error	8	5575.2	696.90		
Total	14	21235.1			

S = 26.40   R-Sq = 73.75%   R-Sq(adj) = 54.05%

Two-way ANOVA: Ductility (cm) versus % SBS, % LMD

Source	DF	SS	MS	F	P
% SBS	2	16496.4	8248.22	11.81	0.004
% LMD	4	3580.8	895.19	1.28	0.353
Error	8	5585.2	698.15		
Total	14	25662.4			

S = 26.42   R-Sq = 78.24%   R-Sq(adj) = 61.91%

Two-way ANOVA: Ductility (Cm) versus % M.Eva, % HOFA

Source	DF	SS	MS	F	P
% M.Eva	2	1644.2	822.08	1.16	0.360
% HOFA	4	11482.2	2870.54	4.07	0.044
Error	8	5649.2	706.15		
Total	14	18775.5			

S = 26.57   R-Sq = 69.91%   R-Sq(adj) = 47.35%

Minitab ANOVA printout: DUCTILITY

Two-way ANOVA: Ductility (Cm) versus % M.Eva, % CKD

Source	DF	SS	MS	F	P
% M.Eva	2	6763.0	3381.51	9.40	0.008
% CKD	4	8825.7	2206.43	6.13	0.015
Error	8	2877.5	359.68		
Total	14	18466.2			

S = 18.97   R-Sq = 84.42%   R-Sq(adj) = 72.73%

Two-way ANOVA: Ductility (Cm) versus % M.Eva, % LMD

Source	DF	SS	MS	F	P
% M.Eva	2	8099.2	4049.61	9.48	0.008
% LMD	4	5197.0	1299.24	3.04	0.085
Error	8	3417.8	427.22		
Total	14	16714.0			

S = 20.67   R-Sq = 79.55%   R-Sq(adj) = 64.21%

Minitab ANOVA printout: PENETRATION

Two-way ANOVA: Penetr (mm) versus % Sulfur, % HOFA

Source	DF	SS	MS	F	P
% Sulfur	3	762.43	254.144	3.47	0.051
% HOFA	4	2274.79	568.697	7.76	0.003
Error	12	879.95	73.329		
Total	19	3917.17			

S = 8.563   R-Sq = 77.54%   R-Sq(adj) = 64.43%

Two-way ANOVA: Penetr (mm) versus % Sulfur, % CKD

Source	DF	SS	MS	F	P
% Sulfur	3	3078.29	1026.10	9.41	0.002
% CKD	4	53.58	13.40	0.12	0.972
Error	12	1309.08	109.09		
Total	19	4440.95			

S = 10.44   R-Sq = 70.52%   R-Sq(adj) = 53.33%

Two-way ANOVA: Penetr (mm) versus % Sulfur, % LMD

Source	DF	SS	MS	F	P
% Sulfur	3	1996.43	665.476	10.56	0.001
% LMD	4	155.36	38.840	0.62	0.659
Error	12	756.16	63.013		
Total	19	2907.95			

S = 7.938   R-Sq = 74.00%   R-Sq(adj) = 58.83%

Two-way ANOVA: Penetration (dmm) versus % Sludge, % HOFA

Source	DF	SS	MS	F	P
% Sludge	2	7.14	3.571	0.24	0.795
% HOFA	4	951.66	237.914	15.76	0.001
Error	8	120.75	15.094		
Total	14	1079.55			

S = 3.885   R-Sq = 88.81%   R-Sq(adj) = 80.43%

Two-way ANOVA: Penetration (dmm) versus % Sludge, % CKD

Source	DF	SS	MS	F	P
% Sludge	2	5498.20	2749.10	16.99	0.001
% CKD	4	907.39	226.85	1.40	0.316
Error	8	1294.70	161.84		
Total	14	7700.29			

S = 12.72   R-Sq = 83.19%   R-Sq(adj) = 70.58%

Minitab ANOVA printout: PENETRATION

Two-way ANOVA: Penetration (dmm) versus % Sludge, % LMD

Source	DF	SS	MS	F	P
% Sludge	2	10184.2	5092.11	98.57	0.000
% LMD	4	917.6	229.40	4.44	0.035
Error	8	413.3	51.66		
Total	14	11515.1			

S = 7.188   R-Sq = 96.41%   R-Sq(adj) = 93.72%

Two-way ANOVA: Penetration (dmm) versus % SBS, % HOFA

Source	DF	SS	MS	F	P
% SBS	2	409.67	204.833	1.64	0.253
% HOFA	4	641.80	160.449	1.28	0.353
Error	8	999.21	124.901		
Total	14	2050.67			

Two-way ANOVA: Penetration (dmm) versus % SBS, % CKD

Source	DF	SS	MS	F	P
% SBS	2	1737.63	868.816	16.41	0.001
% CKD	4	145.81	36.453	0.69	0.620
Error	8	423.60	52.950		
Total	14	2307.04			

S = 7.277   R-Sq = 81.64%   R-Sq(adj) = 67.87%

Two-way ANOVA: Penetration (dmm) versus % SBS, % LMD

Source	DF	SS	MS	F	P
% SBS	2	1487.69	743.843	11.84	0.004
% LMD	4	297.63	74.407	1.18	0.387
Error	8	502.49	62.812		
Total	14	2287.81			

S = 7.925   R-Sq = 78.04%   R-Sq(adj) = 61.56%

Two-way ANOVA: Penetration (dmm) versus % M.Eva, % HOFA

Source	DF	SS	MS	F	P
% M.Eva	2	396.77	198.385	12.36	0.004
% HOFA	4	3476.55	869.138	54.14	0.000
Error	8	128.44	16.055		
Total	14	4001.76			

S = 4.007   R-Sq = 96.79%   R-Sq(adj) = 94.38%

Minitab ANOVA printout: PENETRATION

Two-way ANOVA: Penetration (dmm) versus % M.Eva, % CKD

Source	DF	SS	MS	F	P
% M.Eva	2	556.79	278.394	12.29	0.004
% CKD	4	1268.40	317.099	13.99	0.001
Error	8	181.27	22.659		
Total	14	2006.46			

S = 4.760   R-Sq = 90.97%   R-Sq(adj) = 84.19%

Two-way ANOVA: Penetration (dmm) versus % M.Eva, % LMD

Source	DF	SS	MS	F	P
% M.Eva	2	251.93	125.963	7.56	0.014
% LMD	4	1330.82	332.706	19.98	0.000
Error	8	133.23	16.654		
Total	14	1715.98			

S = 4.081   R-Sq = 92.24%   R-Sq(adj) = 86.41%

Minitab ANOVA printout: VISCOSITY

Two-way ANOVA: viscosity (cP) versus % Sulfur, % HOFA

Source	DF	SS	MS	F	P
% Sulfur	3	3049988	1016663	13.79	0.000
% HOFA	4	21482361	5370590	72.85	0.000
Error	12	884682	73724		
Total	19	25417031			

S = 271.5   R-Sq = 96.52%   R-Sq(adj) = 94.49%

Two-way ANOVA: viscosity (cP) versus % Sulfur, % CKD

Source	DF	SS	MS	F	P
% Sulfur	3	880344	293448	43.61	0.000
% CKD	4	84063	21016	3.12	0.056
Error	12	80750	6729		
Total	19	1045156			

S = 82.03   R-Sq = 92.27%   R-Sq(adj) = 87.77%

Two-way ANOVA: viscosity (cP) versus % Sulfur, % LMD

Source	DF	SS	MS	F	P
% Sulfur	3	812750	270917	55.57	0.000
% LMD	4	37313	9328	1.91	0.173
Error	12	58500	4875		
Total	19	908563			

S = 69.82   R-Sq = 93.56%   R-Sq(adj) = 89.81%

Two-way ANOVA: Viscosity (cP) versus % Sludge, % HOFA

Source	DF	SS	MS	F	P
% Sludge	2	16523187	8261593	3.34	0.088
% HOFA	4	114186173	28546543	11.54	0.002
Error	8	19782531	2472816		
Total	14	150491890			

S = 1573   R-Sq = 86.85%   R-Sq(adj) = 77.00%

Two-way ANOVA: Viscosity (cP) versus % Sludge, % CKD

Source	DF	SS	MS	F	P
% Sludge	2	103113	51557	6.56	0.021
% CKD	4	720312	180078	22.93	0.000
Error	8	62833	7854		
Total	14	886259			

S = 88.62   R-Sq = 92.91%   R-Sq(adj) = 87.59%

Minitab ANOVA printout: VISCOSITY

Two-way ANOVA: Viscosity (cP) versus % Sludge, % LMD

Source	DF	SS	MS	F	P
% Sludge	2	12938	6469.0	3.66	0.074
% LMD	4	261647	65411.7	37.03	0.000
Error	8	14131	1766.4		
Total	14	288716			

S = 42.03   R-Sq = 95.11%   R-Sq(adj) = 91.43%

Two-way ANOVA: Viscosity(cP) versus % M.Eva, % HOFA

Source	DF	SS	MS	F	P
% M.Eva	2	3167615	1583808	8.81	0.009
% HOFA	4	41347783	10336946	57.52	0.000
Error	8	1437585	179698		
Total	14	45952983			

S = 423.9   R-Sq = 96.87%   R-Sq(adj) = 94.53%

Two-way ANOVA: Viscosity(cP) versus % M.Eva, % CKD

Source	DF	SS	MS	F	P
% M.Eva	2	1783090	891545	22.34	0.001
% CKD	4	1160951	290238	7.27	0.009
Error	8	319261	39908		
Total	14	3263301			

S = 199.8   R-Sq = 90.22%   R-Sq(adj) = 82.88%

Two-way ANOVA: Viscosity(cP) versus % M.Eva, % LMD

Source	DF	SS	MS	F	P
% M.Eva	2	602103	301051	36.98	0.000
% LMD	4	413129	103282	12.69	0.002
Error	8	65131	8141		
Total	14	1080363			

S = 90.23   R-Sq = 93.97%   R-Sq(adj) = 89.45%

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